



	Experiment title: In operando study of structural and strain effects in iridium-ruthenium-based catalyst for the anode of the proton exchange membrane water electrolyzers	Experiment number: CH-6568
Beamline: ID31	Date of experiment: from: 28.7. 2023 to: 1.8. 2023	Date of report: 5.9.2023
Shifts: 15	Local contact(s): Dr. Jakub Drnec	<i>Received at ESRF:</i>
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Report:

The aim of beamtime CH-6568 was to study the relationship between the electrochemical performance and complex structural concentration-dependent interplay between Ir and Ru in IrRu mixed catalysts for Oxygen Evolution Reaction (OER) in Proton Exchange Membrane Water Electrolyzers (PEM-WE). Two specific concentrations, the IrRu 1:1 and IrRu 1:3, were examined *in operando* in WAXS/SAXS-compatible PEM-WE single cell. This was motivated by our preliminary measurements which showed that the IrRu 1:3 features better activity and stability than Ir:Ru 1:1 albeit having less Ir.

We measured the samples in form of full Membrane Electrode Assemblies (MEAs) within the PEM-WE cell, first on OCV and then conducted an increasing step-like galvanostatic procedure (from 100 mA cm⁻² to 600 mA cm⁻²) followed by longer stability period at 300 mA cm⁻²; electrochemical impedance spectroscopy was measured periodically. During the electrochemical procedure the catalyst/membrane interface was regularly probed by X-rays (75.5 keV – just below Ir K edge) to track the potential-induced structural evolution of the catalysts. Consequently, Rietveld refinement was employed to track the lattice parameter evolution of Ir and Ru within the alloy and respective crystallite sizes.

The beamtime was successful – we acquired a huge amount of data that is currently being analyzed and prepared for publication. As can be seen from the Fig.1 attached below, the PEM-WE performance of IrRu 1:1 and IrRu 1:3 was nearly identical, even though IrRu 1:3 contains much less Ir. Both IrRu alloys surpassed the pure Ir sample. The preliminarily analyzed X-ray diffraction data (Fig. 2) reveal that Ir and Ru in both systems tend to have strained lattice parameters confirming alloy formation; this effect being arguably more evident in case of IrRu 1:3. Moreover, in sample IrRu 1:3 the hcp phase typical for Ru remains unaltered for the whole duration of the experiment, while in IrRu 1:1 it continuously decreases. Therefore, we believe that the better activity and

stability of IrRu 1:3 stems from the fact that in this concentration, the relatively small amount of Ir is able to stabilize Ru hcp phase or perhaps the Ir itself undergoes the transition to much less common hexagonal structure. We are currently carrying out the *postmortem* XPS and EDX analysis to further complete the picture.

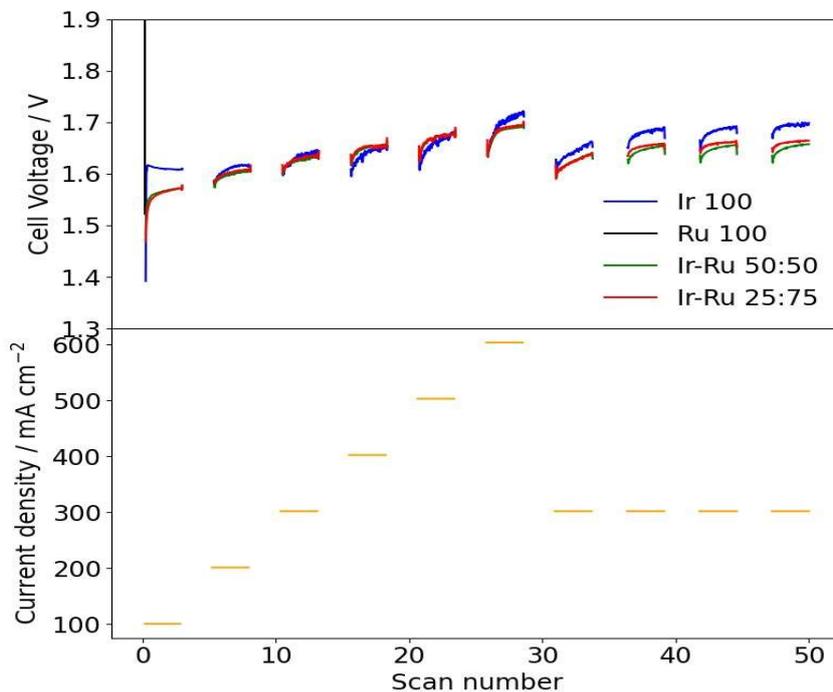


Fig. 1: PEM-WE single cell electrochemical performance of IrRu catalysts

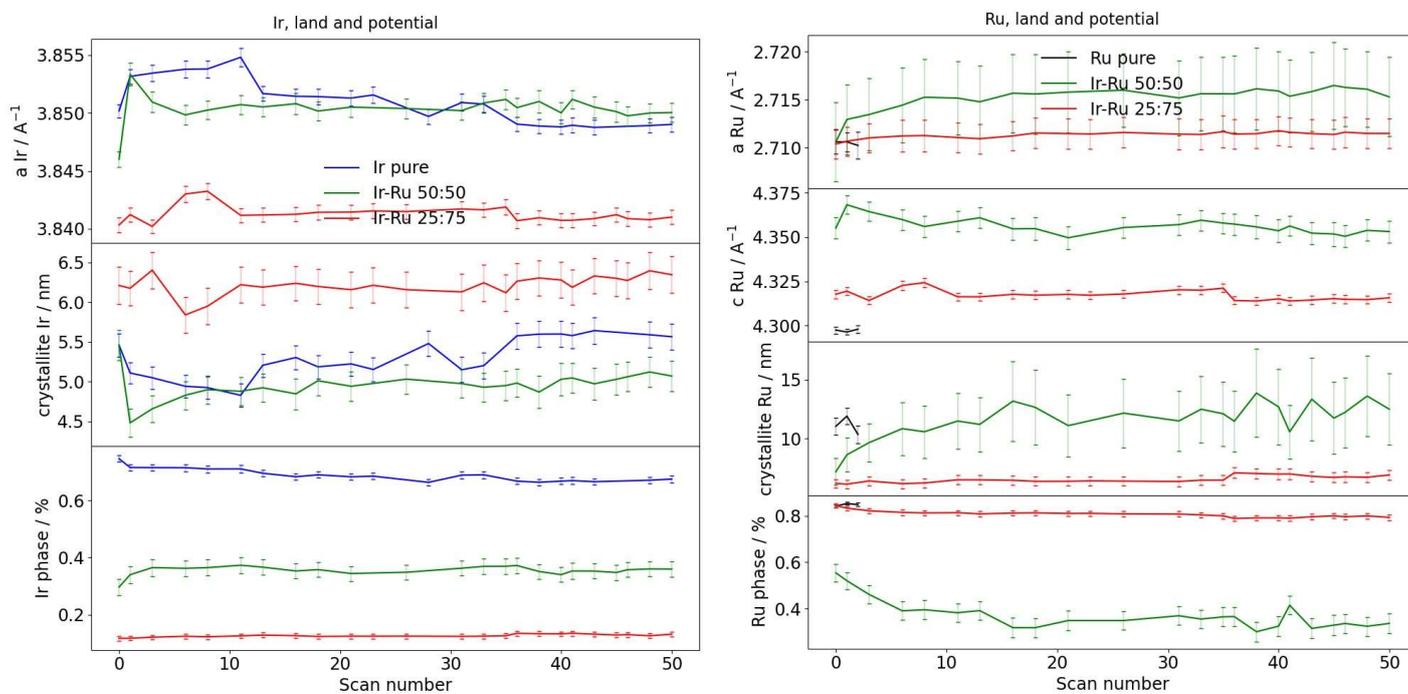


Fig. 2: X-ray diffraction analysis of IrRu catalysts *in operando* (Ir left, Ru right)