## Degradation Mechanisms of Oxygen Evolution Reaction Electrocatalysts: A Combined Identical-Location Transmission Electron Microscopy and X-Ray Photoelectron Spectroscopy Study

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Determining the degradation mechanism of oxygen evolution reaction (OER) catalysts is fundamental to design improved polymer electrolyte membrane water electrolyser (PEMWE) devices. In this study, we combined identical-location transmission electron microscopy, X-ray photoelectron spectroscopy and inductively coupled plasma mass spectrometry to determine the changes in morphology and composition of iridium nanocatalysts covered by a thin oxide layer ( $IrO_x$ ) in simulated PEMWE anode operating conditions. The results show that iridium valency increased rapidly during a conditioning step and in a softer manner after electrolysis. The catalysts containing initially metallic iridium were composed of a mix of Ir(III), Ir(IV) and Ir(V) after OER conditions while catalysts containing initially Ir(IV) solely had the same composition after OER. IL-TEM images show that Ir dissolution/redeposition and particle detachment occurred during electrochemical conditioning while agglomeration/coalescence and dissolution were observed after electrolysis. Iridium dissolution was quantified after a constant current electrolysis and we observed a direct relationship between the proportion of Ir(IV) in the catalysts and their stability in OER conditions.



50 k cycles between

1.2 and 1.6 V vs. RHE



Figure 1: ILTEM images of  $IrO_x$  nanoparticles supported on antimony-doped tin dioxide aerogel before/after 50 k potential cycles between 1.2 < E < 1.6 V vs. RHE.

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## REFERENCES

1. F. Claudel, L. Dubau, G. Berthomé, L. Sola-Hernandez, C. Beauger, L. Piccolo, F. Maillard, submitted.