ECATALYTIX II

April 8-10 2026

Strasbourg

Collège Doctoral Européen

Book of Abstracts

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  46 Boulevard de la Victoire, 67000 Strasbourg

- **Gala Dinner :** Le Meteor
  10, rue du 22 Novembre, 67000 Strasbourg

Wednesday, April 8th

Session	Time	Activity
	09:00	**Welcome**
(#1) Recent Developments in Operando Electrocatalysis	09:10	**IT1 - Elena R. Savinova** 'Chasing Reaction Intermediates with Operando Spectroscopies'
	09:50	**OC1 - Meryem Ennaji** 'Stabilization of IrCu Aerogel Catalyst for PEMWE from Operando X-ray Total Scattering-PDF Analysis to Flash Heat Treatments'
	10:10	**OC2 - Cem Celikutku** 'Tracking Electronic Structure Evolution in Molecular Iron Catalysts by Operando Fe K-edge XANES'
	10:30	**Coffee break**
(#1) Recent Developments in Operando Electrocatalysis	11:00	**ET1 - Andrey Shavorskiy** - 'Kinetics of Electric Double Layer Formation Studied by *Operando* Dip-and-Pull APXPS'
	11:30	**OC3 - Kenneth Crossley** 'Examining Local Structure-Activity Relations in Identical Metal Composition OER Catalysts with *Operando* X-ray Techniques'
	11:50	**OC4 - Nicolò Orshinger** 'Operando Spectroscopy Of Redox Dynamics In Hematite/NiMo Photoanodes'
	12:10	**OC5 - Julie Guehl** 'From Cradle to Grave – a Venture into The Life of Nickel Nanoparticles as Hydrogen Evolution Reaction Electrocatalysts'
	12:30	**Lunch break**
(#1) Theoretical Approaches in Operando Electrocatalysis	14:00	**IT2 - Katarina Doblhoff-Dier** 'Exploiting Synergies Between Modeling and Experiments to Decipher the Structure of the Electric Double Layer'
	14:40	**OC6 - Cecilia Irène Gho** 'Understanding Electrochemical Response of Operando Liquid-Phase TEM Cell Through Multiphysics Modelling'
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	15:30	**Round table #1 - Bridging Theory and Experiments in Operando Electrocatalysis**
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Thursday, April 9th

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(#3) At the Frontiers Operando Electrocatalysis	09:10	**IT3 - Jakub Drnec** 'Reliable, Reproducible and Automated *Operando* Electrocatalysis Methodologies at Fourth-Generation Synchrotrons'
	09:50	**OC7 - Paul Chassagne** 'Mapping the pH in the Local Reaction Environment using *Operando* Fluorescence Confocal Microscopy'
	10:10	**OC8 Lars Pötters -** 'Understanding The Urea Oxidation Reaction on Nickel Oxides by Electrochemistry and *In Situ* Photocurrent and Reflectivity Measurements'
	10:30	**Coffee break**
(#3) At the Frontiers Operando Electrocatalysis	11:00	**ET2 - Bruna Ferreira Gomes** 'Probing Competitive Adsorption and Site Blocking in Electrocatalysis by *Operando* $\Delta\mu$ XANES and FEXRAV'
	11:30	**OC9 - R. E. Wibowo** 'The Effect of O2 on the Cathodic Electrochemically Mediated Amine Regeneration Processes via *In Situ* Soft and Hard X-ray Absorption Spectroscopy'
	11:50	**OC10 - Petra Khater** 'Hydrogen-Driven Phase Transitions in Palladium Nanoparticles Revealed by *In Situ* BCDI'
	12:10	**OC11 - Alba Garzón Manjón** 'Insights into the Degradation of Metallic Core-Shell Nanoparticles under Fuel Cell Conditions by 3D Identical Location STEM'
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(#4) Lab-Scale Operando Electrocatalysis	14:00	**IT4 - Janis Timoshenko** 'Unveiling the Heterogeneous Structures of Electrocatalysts with Synchrotron- and Lab-Based *Operando* XAS and Machine Learning'
	14:40	**OC12 - Tim Welmers** 'Ionomer-Electrocatalyst Interactions for Fluorine Free Ionomers on Pt Electrodes Using ORR *Operando* ATR-SEIRAS'
		OC13 - Angelica Chiodoni 'Nanostructured Palladium Electrodes for Reliable Electrochemistry in Liquid-Phase TEM'
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Session	Time	Activity
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	09:50	**OC15 - Nathaly Ortiz Peña** 'Hydrogen Evolution Reaction on Pt Nano-Aggregates by Correlative Multiscale Microscopies'
	10:10	**OC15 - L. Moriau** 'Investigation of IrO2 and Ir Electrocatalysts During OER by *In Situ* Raman Spectroscopy'
	10:30	Coffee break
(#4) Lab-Scale Operando Electrocatalysis	11:00	**ET3 - Raphaël Chattot** 'Primacy of Lattice Distortion over Strain in Precious Nanoalloys for Oxygen Electrocatalysis'
	11:30	**OC17 - Marc F. Tesch** 'Combination of Laboratory- and Synchrotron-Based Potentiodynamic Techniques - Opportunities and Challenges'
	11:50	**OC18 - Michal Ronovsky** 'From Artefacts to Insights: Challenges and Opportunities in *Operando* NAP-XPS and XRD of IrOx Catalyst Layers in PEMWE'
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Chasing Reaction Intermediates with Operando Spectroscopies

Savinova, Elena[1]

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Understanding reaction intermediates in heterogeneous electrocatalysis has become a central challenge in modern electrochemistry, driving the rapid development and application of diverse operando spectroscopic techniques. In this lecture, we critically examine whether identifying such intermediates is truly essential for advancing the field, to what extent their detection is experimentally feasible, and under which conditions meaningful insights can be obtained.

Drawing on selected examples from our work alongside contributions from the literature, we highlight the capabilities and limitations of various operando spectroscopies. We also demonstrate that complementary electrochemical methods - often deceptively simple - can yield equally valuable mechanistic information.

Particular attention will be given to the challenges of interpreting operando data, including the distinction between reactive intermediates and non-reactive surface species. Finally, we discuss the broader relevance of operando studies and evaluate how the insights they provide can be effectively leveraged to guide the rational design of improved electrocatalytic materials.

Exploiting synergies between modeling and experiments to decipher the structure of the electric double layer

K. Doblhoff-Dier[1], Lucas de Kam[1], Justina H. Moss[1], Rick S. Kort[1]

Collaborators:
Mariana C. O. Monteiro[1,2], Marc T. M. Koper[1], Sheena Louisia[3], Esther Alarcon-Llado[4], Daphne Antony[4], Alfred Larsson[1], Rik Mom[1]

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The structure of the electric double layer at electrified interfaces plays a pivotal role in governing key electrochemical reactions. Understanding this structure requires advanced operando techniques. Important insight into the double layer structure can, for example, be obtained from X-ray-based techniques, electrochemical approaches, and scanning probe microscopy. More detailed understanding, however, can often be achieved when coupling these experimental approaches to complementary, computational techniques, including analytical modeling, DFT modeling, molecular dynamics studies and machine learning approaches.

In this talk, I will illustrate how the synergy of experimental and theoretical approaches can enhance our understanding of the double layer structure, beyond what can be achieved by theory or experiment alone. Examples will include (i) how ab-initio and force-field molecular dynamics simulations complement X-ray truncation rod measurements by revealing the solvation structures around the ions that are challenging to probe experimentally; (ii) how analytic double layer models help rationalizing XAS data for electrochemical interfaces in micro-porous systems and XPS data obtained in dip-and-pull experiments; and, if time allows, (iii) how modeling and MD can provide an interpretation of liquid AFM measurements.

Reliable, Reproducible and Automated Operando Electrocatalysis Methodologies at Fourth-Generation Synchrotrons.

J. Drnec[1] and M. Ennaji[2] and R. Chattot[2]

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[2] ICGM, Univ. Montpellier, CNRS, ENSCM, 34095 Montpellier, France

Operando synchrotron characterization is now the workhorse in electrocatalysis, providing deep mechanistic understanding across unprecedented spatiotemporal scales. The transition to fourth-generation light sources has enabled high-throughput measurements with better resolution, yet this data-rich era introduces critical challenges regarding data fidelity and the management of massive datasets. Currently, a significant bottleneck exists as only a fraction of the terabytes acquired at beamlines is fully analyzed or exploited. This contribution proposes strategies to address these reliability and quantity issues through standardized quality-assessment workflows and machine-learning-driven analysis.

Data fidelity is paramount for reliable research and requires a rigorous evaluation of beam-interaction effects. The extreme intensity of modern X-ray beams can trigger the radiolysis of electrolyte species, generating radicals that fundamentally alter chemical kinetics and mechanical integrity. To ensure the X-ray probe does not bias the observed chemistry, we propose systematic benchmarking to evaluate dose-dependent structural and chemical changes. In general, we advocate for a holistic assessment based on reliability, representativeness, and reproducibility, supported by standardized metadata ontologies to ensure datasets that can be used in further research and are AI-readable, avoiding "garbage-in-garbage-out" scenarios [1].

To manage the data quantity challenge, we move beyond computationally heavy inverse algorithms by demonstrating a real-time data treatment approach using Convolutional Neural Networks (CNN) for automated Rietveld analysis [2]. Our methodology [3] utilizes a hybrid training strategy, combining simulated physical models with Rietveld pre-analyzed experimental datasets. This approach allows the network to learn from idealized models while remaining robust against the noise and nuances of real-world operando data. Such automated pipelines are essential to accelerating the "data-to-end-result" speed, ensuring high-throughput experimental capabilities are matched by reliable, high-speed analysis.

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3. M. Ennaji, R. Chattot, J. Drnec, unpublished

Unveiling the Heterogeneous Structures of Electrocatalysts with Synchrotron- and Lab-based Operando XAS and Machine Learning

Janis Timoshenko

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Unraveling the structure-property relationships is one of the open problems in studies of functional nanomaterials, and heterogeneous electrocatalysis in particular. One of the reasons for this is the dynamic transformation of the electrocatalyst's active species on different time- and length-scales under working conditions, which are hard to account for using available theoretical approaches, and are also often hard to predict from the ex-situ measurements of the as-prepared pre-catalysts. X-ray absorption spectroscopy (XAS) is a premier technique for tracking the structure of working functional materials due to its sensitivity to atomistic and electronic structure of the material, element specificity, and applicability to a broad range of different samples and experimental conditions [1]. Nonetheless, the interpretation of XAS data often remains challenging, and is particularly difficult, if the investigated material is a mixture of different species, and/or evolves in time in the course of the reaction.

Recent advances in *operando* XAS provide a solution to this problem. On one hand, lab-based XAS now provides the possibility to screen large parameter spaces and design control experiments, in particular, long-term studies that are incompatible with the traditional synchrotron beamtime access mode [2]. On the other hand, the recent developments in data science methods enable the interpretation of complex datasets containing contributions from multiple coexisting species. In particular, the development of unsupervised and supervised machine learning methods for the interpretation of X-ray absorption near edge (XANES) and Extended X-ray Absorption Fine Structure (EXAFS) spectra provides key insight for understanding the evolving structures of complex heterogeneous materials [3-6].

Here we demonstrate the potential of lab-based XAS and machine learning-based data analysis on an example of studies of working catalysts for CO_2 electrocatalytic reduction [2,4] and oxygen evolution in alkaline electrolytes [6]. The compelling evidence about the nature of the catalyst active states, obtained from operando time-resolved XAS coupled with advanced data analysis approaches allows us to decouple the contributions of different species coexisting in working catalysts to the catalysts' functionality and provides guidelines for the further optimization of catalytic systems and reaction protocols.

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Cryogenic and Liquid-Phase Electron Microscopy for Electrocatalytic Materials

Vasiliki Tileli

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Understanding catalyst surface and interface interactions is essential for designing more efficient energy systems. In this talk, I will discuss how advanced electron microscopy techniques such as liquid-phase transmission electron microscopy (TEM) and cryogenic electron tomography are used to gain valuable insights into complex organic/inorganic and solid/liquid/gas interfaces. The presentation will cover three specific systems. First, I will review advancements in confined TEM microcells for the real-time imaging of copper catalysts during the carbon dioxide reduction reaction (CO_2RR). I will present results that enable the study of these processes in situ, specifically focusing on how the metallic/oxide surface layer of copper nanocubes influences the mechanisms responsible for their evolution during the initial stages of CO_2RR[1]. Second, I will discuss how electrochemical liquid-phase TEM on cobalt-based oxygen-evolving oxides can reveal the effects of electrowetting and active Co^{2+}/Co^{3+} redox transition as surfaces shift toward a hydrophilic character [2]. Finally, I will demonstrate how cryogenic electron tomography preserves the three-dimensional structure of proton exchange membrane fuel cell (PEMFC) cathode components, enabling the precise measurement of catalyst accessibility [3].

Overall, these advanced electron diagnostics offer fundamental insights into the "true" starting state of catalytic systems and their evolution during electrocatalytic processes, serving as a powerful complement to other characterization techniques.

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Elected Communications

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Toward a Statistical Approach to Electrocatalysis *via In Situ* and *Operando* High-Energy Powder X-Ray Scattering

Amir Gasmi [1], Meryem Ennaji [1], Jakub Drnec [2] and Raphaël Chattot [1, *]

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Our understanding of catalytic processes still largely relies on simplifying assumptions regarding the structure of active sites, often described as homogeneous entities in terms of coordination, strain, and composition at the nanoscale. However, nanomaterials cannot be regarded as simple miniaturized versions of single crystals: they exhibit a high degree of site heterogeneity, local fluctuations in structure and composition, and dynamic states that depend on operating conditions. Their catalytic properties therefore emerge from an intrinsic complexity that is difficult to capture within idealized models, and incompatible with limited computational resources. Thus, the very notion of the “active site” appears largely as a conceptual mirage, inherited from studies on model surfaces and single crystals, assuming a static and deterministic view of catalytic materials.

Rather than attempting to identify a single, or few well-defined active site(s), it becomes necessary to adopt a statistical approach to electrocatalysis, in which activity arises from a dynamic distribution of local configurations. This perspective calls for new descriptors aimed at quantifying fluctuations and heterogeneities around average values of usual parameters. Such statistical descriptors, accessible in particular through advanced *in situ* and/or *operando* approaches using physical or electrochemical probes, represent a promising pathway toward establishing more realistic relationships between material structure and catalytic performance under operating conditions.

In this talk, we first expose this principle in the case of PtNi nanoalloys catalysts for the oxygen reduction reaction across critical stages of their development (*viz.* as-synthesized, post-electrochemical activation, after membrane electrode assembly fabrication, and following accelerated stress testing) using a comprehensive set of *ex situ*, *in situ*, *operando*, and *post mortem* characterization techniques, notably high-energy X-ray scattering, which allows assessing the contributions from alloying and structural effects. Our results reveal that local lattice distortion, rather than global strain and ligand effects, is an important factor effectively contributing to both catalytic activity and durability in PEMFC [1–4]. The above conclusions are then tentatively extended to iridium-based nanoalloys for the oxygen evolution reaction in proton exchange membrane water electrolysers (PEMWEs) [5].

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Probing Competitive Adsorption and Site Blocking in Electrocatalysis by Operando Δμ XANES and FEXRAV

Bruna Ferreira Gomes[1], Rameshwori Loukrakpam[1], Tomas Bystron[2], Martin Prokop[2], Karel Bouzek[2], Laia Capdevila Ibanez[1], Christina Roth[1]

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[2]Department of Inorganic Technology, University of Chemistry and Technology Prague, Technicka 5, 16628, Prague 6, Czech Republic.

Understanding adsorption dynamics and site-blocking phenomena under realistic electrochemical conditions remains a central challenge in electrocatalysis. In this work, we present an operando X-ray absorption spectroscopy (XAS) framework that combines differential XANES (Δμ XANES) with time-resolved fixed-energy X-ray absorption voltammetry (FEXRAV) to probe competitive adsorption and transient surface processes on electrocatalyst interfaces.

Building on our recent study on Pt nanoparticles, Δμ XANES is shown to provide semi-quantitative insight into the competitive adsorption of H-, CO-, O- and phosphate-derived species, enabling the correlation of spectral intensity with relative surface coverage despite the intrinsic bulk sensitivity of hard XAS. When coupled with FEXRAV, real-time tracking of adsorption and removal processes becomes possible, allowing us to directly follow CO stripping, hydrogen re-adsorption, and site blocking by H_3PO_3 under dynamic electrochemical operation. This combined approach reveals how phosphate-derived impurities strongly compete for Pt active sites, suppressing hydrogen and oxygen adsorption even at low concentrations, with direct implications for high-temperature PEM fuel cells and related technologies[1-3].

We further extended this operando concept through recent experiments at DESY (PETRA III, P65), where fixed-energy XAS was applied to Cu-based catalysts during pulsed CO_2 electroreduction. By synchronizing electrochemical pulses with fluorescence-detected XAS at a single energy, oxidation-reduction dynamics between copper species were resolved with sub-second time resolution. FEXRAV measurements proved highly effective for capturing transient surface phenomena while minimizing beam damage.

Overall, these results demonstrate that the combination of Δμ XANES and FEXRAV provides a powerful operando toolbox to link steady-state surface speciation with fast adsorption dynamics. The methodology is broadly applicable to fuel cells, electrolyzers, and CO_2 reduction systems, offering new opportunities to unravel competitive adsorption and site-blocking phenomena under realistic, time-dependent operating conditions.

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Kinetics of Electric Double Layer Formation Studied by Operando Dip-and-Pull APXPS

Andrey Shavorskiy, Alexander Yaroslavtsev, Robert Temperton

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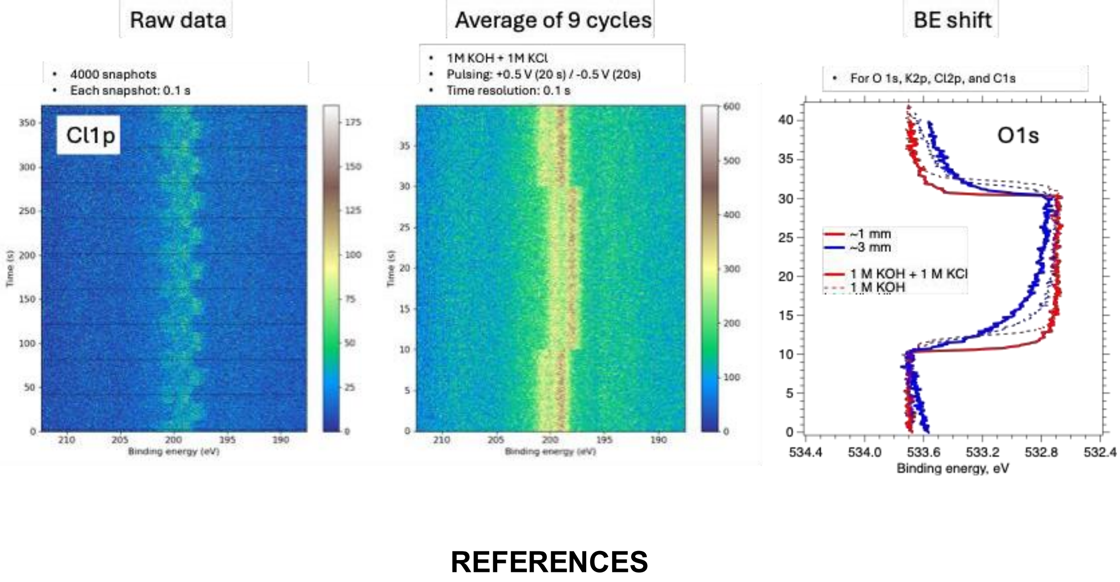
Dip-and-pull (also known as "meniscus") Ambient Pressure XPS has been increasingly popular in studying solid-liquid interfaces of a wide range of electrochemical systems, such as, e.g., batteries, electrocatalysts, or corroding alloys. Its popularity roots in the method's high surface sensitivity, chemical specificity, non-contact nature of the probe, and intrinsic in situ / operando capabilities. Despite its limitations, which have been carefully examined in several recent studies,[1-6] the method is ideal for investigating the fundamental properties of the interfacial double layer, of both capacitive and faradic nature.

In this study, we examined the influence of the ion composition of the electrolyte on the capacitive properties of EDL by employing the time-resolved APXPS and chronoamperometry during modulation of the sample bias. A time evolution of the local potential in the form of photoemission peak shifts has been measured with 0.1 s time resolution from 1M KOH with and without 1M KCl at different distances from the bulk electrolyte after changing the applied potential by 1.0 V (+/- 0.5 V vs OCV). 9 such modulations have been averaged to improve the signal-to-noise ratio. As expected, the time constant for photoemission peak shift by 1.0 eV (equal to the potential jump) increases with the increase of the distance from the bulk electrolyte. This trend holds true for KOH and KOH + KCl electrolytes. However, in the case of pure KOH, the time constants are significantly smaller than when the chloride is added. We employ the meniscus model developed by NN to explain the observed trends and discuss the implications for the electrocatalysis.



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Oral Communications

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Stabilization of IrCu Aerogel Catalyst for PEMWE from Operando X-ray Total Scattering-PDF analysis to Flash Heat Treatments

Meryem Ennaji* [a], Amir Gasmi [a], Morgane Stodel [b], Frédéric Jaouen [a],
Jakub Drnec [c], Raphaël Chattot [a]

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Proton exchange water electrolyzer (PEMWE) is rising up as an advanced and effective solution for green hydrogen production [1]. Green hydrogen, offers an alternative to fossil fuel, providing flexible energy storage for extended periods and enabling highly efficient reconversion to electricity through fuel cells. However, its market penetration is still limited by two key challenges: (1) scarcity of iridium (Ir)-based anodes required to catalyze the sluggish oxygen evolution reaction (OER), and (2) the limited understanding of PEMWE performance and durability, which slows technological advancement. Nanostructured and unsupported Ir-based catalysts, which maximize Ir utilization, have demonstrated promising performance [2], however, the nature of their active site during OER and the mechanism driving their deactivation remain unknown.

In this study, operando X-ray total scattering combined with atomic pair distribution function (PDF) analysis is employed to probe both crystalline and amorphous atomic structures of nanostructured, unsupported Ir-based catalysts in a liquid cell (model system) and in a PEMWE, representative of near real world operating conditions [3].

The results provide insights into the complex, potential-dependent local structure dynamics occurring during PEMWE operation and reveal key stability limitations of the IrCu aerogel catalyst. To address these challenges, flash heat treatment is explored as a promising strategy to achieve an optimal balance between catalytic activity and structural stability [4], specifically targeting the degradation mechanisms identified through accelerated stress testing (AST) in PEMWE.

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Tracking Electronic Structure Evolution in Molecular Iron Catalysts by Operando Fe K-edge XANES

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Iron phthalocyanines and porphyrins are benchmark molecular catalysts for electrochemical CO_2 reduction and related reactions, where small changes in the Fe electronic structure can strongly impact activity and selectivity. *Operando* X-ray absorption spectroscopy provides direct access to these changes by tracking the Fe K-edge XANES response under applied potential, including edge position shifts, white-line evolution, and subtle changes in the pre-edge region associated with metal-ligand hybridization and symmetry breaking.

In this contribution, we present an operando Fe K-edge XANES study designed to monitor potential-dependent electronic structure evolution of FeN_4 macrocyclic catalysts under working electrochemical conditions. Measurements were performed at the ASTRA beamline (SOLARIS synchrotron, Poland) using a spectroelectrochemical flow cell, enabling controlled acquisition under catalytic bias in relevant electrolytes and gas environments. The analysis focuses on the full Fe K-edge XANES region, including edge shifts and white-line evolution, while the pre-edge is used as a complementary probe of local symmetry and metal-ligand covalency.

The operando strategy builds on recent ex situ Fe K-edge XAS investigations and calibrated TD-DFT simulations that established a reliable interpretation framework for near- edge features in iron phthalocyanine and porphyrin systems. Combining operando XANES with this validated analysis pipeline aims to distinguish predominantly electronic changes from potential-induced structural distortions and interfacial effects, thereby providing molecular-level insight into active states during operation.

Overall, this work targets mechanistic structure-function relationships in molecular electrocatalysis and contributes transferable spectroscopic descriptors for rational design of hybrid molecular catalyst architectures.

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Examining local structure-activity relations in identical metal composition OER catalysts with *operando* X-ray techniques

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Transition metal (hydr)oxide oxygen evolution reaction (OER) catalysts have the potential to enable a more economical global hydrogen economy if greater activity can be stabilized. The amorphous forms of Co/Ni/Fe based oxide catalysts often deliver greater OER activity than their crystalline analogues[1], but the amorphous activity descriptors are poorly understood. $CoSn(OH)_6$ hydroxide perovskites demonstrate large increases in activity (>50%) during electrochemical activation. Moreover, an amorphous oxide can be created from the same co-precipitation batch by a mild thermal treatment.[2] Using identical metal composition crystalline, crystalline defective, and bulk amorphous catalysts derived from the same $CoSn(OH)_6$ batch, we examine the effect of structure on OER activity. Based on our electrochemical data, we demonstrate that an amorphous structure can be less effective for enhancing activity than surface oxygen vacancies. We also present *operando* and *ex situ* X-ray absorption spectroscopy and diffraction evidence for local ordering of an amorphous catalyst in the first two hours of OER operation. Furthermore, we show that Fe doping can overpower these effects. We expect these results may guide future efforts towards definitive surface engineering of transition metal oxide OER catalysts.

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Operando Spectroscopy Of Redox Dynamics In Hematite/NiMo Photoanodes

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Nickel-based (oxy)hydroxides are among the most used electrocatalysts for alkaline oxygen evolution, and their catalytic activity is intrinsically linked to dynamic redox process that occur under applied bias[1]. In photoelectrochemical systems, these redox processes are influenced by photogenerated charge carriers from the semiconductor substrate too, making static characterization insufficient[2]. Operando techniques capable of tracking oxidation-state changes during operation are required to understand the interplay between semiconductor, cocatalyst, and electrolyte[2].Titanium-doped hematite (Ti:α-Fe_2O_3) photoanodes functionalized with nickel-molybdenum (NiMo) thin films are investigated in alkaline electrolyte (NaOH 0.1 M) by combining operando visible transmission spectroscopy and operando X-ray Absorption Spectroscopy (XAS). The study focuses on tracking the dynamics between Ni^{2+}/Ni^{3+} species (nickel hydroxide $Ni(OH)_2$ and nickel oxyhydroxide NiOOH, respectively) during photoelectrochemical operation. Operando visible transmission measurements, implemented as Static Visible Absorption Spectroscopy and Fixed Wavelength Transmission Voltammetry, probe potential- and light-induced changes in optical transmittance associated with nickel oxidation. Differential Absorbance spectra shows that the 550-650 nm range provide a sensitive probe of the $Ni(OH)_2$/NiOOH redox couple, allowing the tracking of oxidation and reduction processes during cyclic voltammetry under dark and illuminated conditions. These measurements reveal that illumination induces an earlier onset of nickel oxidation at intermediate anodic potentials, as well as modified reduction pathways upon reverse scans. Operando XAS, performed at the Ni K-edge using Fixed Energy X-ray Absorption Voltammetry (FEXRAV), provides element-specific confirmation of these trends. XAS directly tracks changes in the nickel oxidation state and demonstrates that photogenerated holes from hematite actively participate in the oxidation of nickel species at intermediate potential. At higher anodic potentials, XAS reveals an inversion of the light-induced effect, with illumination leading to a more reduced nickel steady state compared to dark conditions, indicating a complex, potential-dependent coupling between charge transport and catalyst redox chemistry. While visible transmission techniques provide a rapid and sensitive probe of global redox- driven optical changes, XAS offers element-specific insight into the underlying oxidation-state evolution. The consistent observation of light-induced early oxidation at intermediate potentials and photoinduced reduction at higher potentials provides key information on the non-trivial role of photogenerated carriers during cocatalyst redox behavior.

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From Cradle To Grave – A Venture Into The Life Of Nickel Nanoparticles As Hydrogen Evolution Reaction Electrocatalysts

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Ni/NiO_x heterostructures are among the most promising non-noble metal electrocatalysts for the hydrogen evolution reaction (HER) in alkaline media. Although the role of chemical composition, particularly the Ni/NiO_x ratio, on the HER activity has been widely studied[1], structure-activity relationships remain poorly understood.Here, we employ an *operando* multiscale X-ray scattering approach to track the evolution of the crystallographic properties of Ni-based electrocatalysts throughout their entire lifecycle, from their formation through electrodeposition, to their use as HER electrocatalysts, and ultimately long-term aging. Specifically, using combined wide- and small-angle X-ray scattering (WAXS and SAXS, respectively), we monitor the formation of Ni nanoparticles (NPs), their oxidation into Ni/NiO_x heterostructures, and their structural and chemical evolution under HER conditions (*e.g.,* nickel hydride formation[2], adsorption and desorption of reactive intermediates, *etc.*).

Our results reveal that, despite variations in the electrodeposition parameters and electrodeposited charge, the Ni crystallite size growth remains broadly comparable across the samples (**Fig. 1**), whereas the nanoparticle size increases significantly. We propose a growth mechanism involving primary nucleation on the carbon support, followed by secondary nucleation on Ni sites rather than on carbon, owing to the lower overpotential for metal-on-metal compared to metal-on-carbon nucleation[3]. We investigate the evolution of Ni particle crystallography under applied potential in 0.1 M KOH across Ni/NiO_x redox transitions and the HER. Overall, this work directly links structural dynamics to electrochemical response, offering new insights for the rational design of durable non-noble HER electrocatalysts.

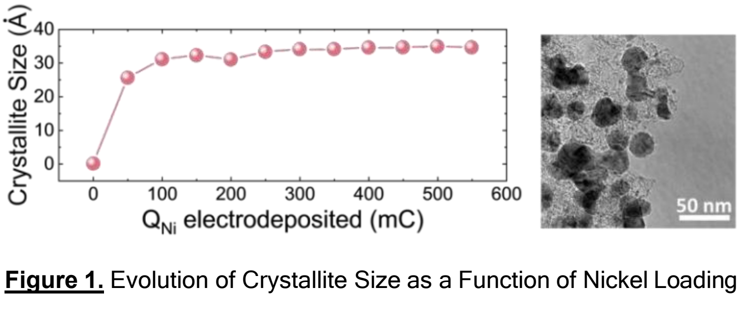

Figure 1. Evolution of Crystallite Size as a Function of Nickel Loading

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Understanding Electrochemical Response of Operando Liquid-Phase TEM cell through Multiphysics Modelling

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Operando electrochemical techniques are essential for elucidating reaction mechanisms and the relationship between structure and electrochemical activity of catalysts under realistic working conditions. Among these, electrochemical liquid-phase transmission electron microscopy (EC-LPTEM) provides unique access to nanoscale morphological, structural, and chemical changes during electrochemical operation[1]. However, the highly confined geometry required for electron transparency fundamentally alters mass transport, potential distribution, and current response, often hindering direct comparison between EC-LPTEM measurements and conventional lab-scale electrochemistry[2]. Despite the growing interest in this technique and urgency of understanding the so-called attenuated electrochemistry[2] observed in EC-LPTEM cells, the literature still lacks clear guidelines describing the behavior and intrinsic limitations of these systems. In this work, the influence of geometric confinement on the electrochemical response of EC- LPTEM cells is investigated using finite element simulations. 2D and 3D models at increasing complexity were developed in COMSOL Multiphysics[3] to account for mass transport and electric field distribution. These models enable the simulation of time-dependent electrochemical experiments, including chronoamperometry and cyclic voltammetry, under realistic cell geometries, electrolyte concentrations, and applied potentials. By systematically varying key parameters, the models decouple the effects of electrolyte confinement on mass transport regimes and electric field configuration, and their combined impact on the electrochemical response. The simulations reveal that the presence of a narrow liquid nanochannel in front of the working electrode induces a non-classical diffusion regime. Moreover, geometric confinement significantly increases the intrinsic resistance of the cell, leading to an attenuated electrochemical response and to non-uniform potential and current density distributions across the working electrode. These effects become increasingly pronounced with stronger electrolyte confinement, underscoring the critical role of cell design in operando electrochemical measurements. Overall, this study provides practical guidelines for the design of operando EC-LPTEM experiments and correct interpretation of the electrochemical data acquired in confined cells. More broadly, the modelling framework offers a versatile platform for the rational design of improved cell geometries and electrode configurations, supporting the development of next- generation operando TEM platforms that enable more reliable comparison with lab-scale electrochemical systems.

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Mapping the pH in the Local Reaction Environment using *Operando* Fluorescence Confocal Microscopy

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In order to properly assess the stability and efficiency of an electrocatalyst, one must know its real, rather than hypothesized, operating conditions. Indeed, as the electrochemical reactions of the H_2 cycle always involve either H^+ or OH^-, they are thus greatly influenced by pH variations. Herein, we developed a technique allowing for the 3D *operando* monitoring of the pH in the local reaction environment (*i.e.*, the immediate vicinity of the electrode) with high spatial and temporal resolutions. It combines the use of confocal laser scanning microscopy (**Fig. 1**) and a new family of pH-sensitive, ratiometric, fluorescent probes. Using this technique, we were able to map the pH changes in the vicinity of an electrode with sub- micrometric and sub-second precision during H^+/OH^- generating/consuming electrochemical reactions in both alkaline and acidic conditions.

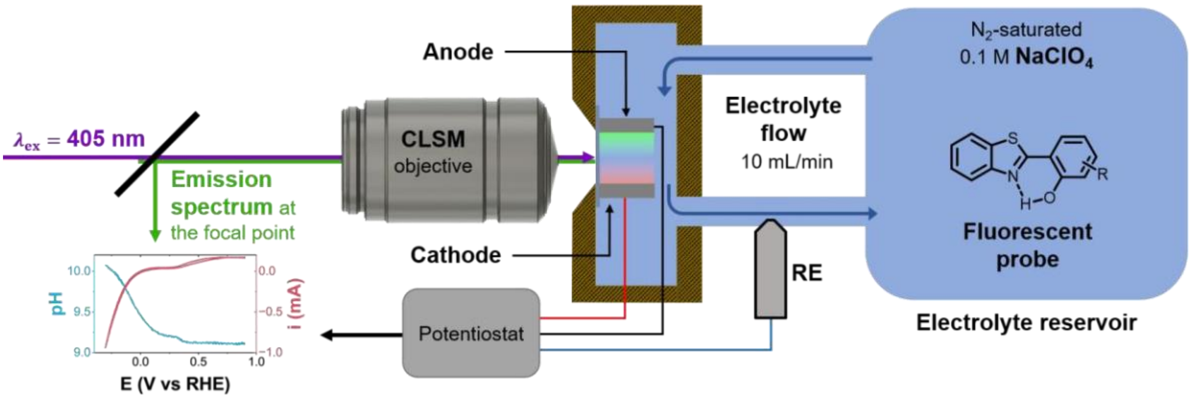

Figure 1 – Scheme of the developed *operando* method. The surface of the working electrode is imaged by the objective of the confocal laser scanning microscope while being under applied potential. An electrolyte containing a pH-sensitive fluorescent probe circulates in the cell. The microscope scans the local reaction environment in the three spatial dimensions in order to map the light emitted by the probe as a function of the applied potential, allowing us to determine the local pH.

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Understanding The Urea Oxidation Reaction on Nickel Oxides by Electrochemistry And In-situ Photocurrent and Reflectivity Measurements

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The electrochemical urea oxidation (UOR) is important in many different fields, e.g. in the remediation of urea-rich effluents in agriculture and industry, and as a stable and non-toxic hydrogen carrier in sustainable energy systems.[1,2] Nickel materials have shown to be the only catalyzing species to overcome the rather sluggish reaction kinetics of UOR. On the complex nickel surface in alkaline media, involving four different oxides (α-/β-Ni[2+]$(OH)_2$ and β-/γ-Ni[3+]OOH), mainly two different types of mechanism for the UOR are discussed, a direct pathway using the Ni[3+] as a catalyst and an indirect one, where a reduction and re-oxidation of the Ni[3+] oxide is a side reaction.[3-5] The electrooxidation of Ni[2+] and urea happens to appear in the same potential range, leading to an overlapped oxidation peak in CV measurements. To distinguish between these processes, we used in-situ photocurrent and reflectivity measurements and complementary electrochemistry, also in a urea-free system.

In a CV-reduction peak analysis, we found that the potential-dependent changing surface species is the γ-Ni[3+]OOH oxide, with an increasing amount with increasing potential for urea-free solution and not increasing for urea-containing electrolyte. We thereby highlight the high importance of understanding the geometry and formation of γ-Ni[3+]OOH for catalyst design. Combining this result with the earlier mentioned in-situ characterizations, we claim that the UOR follows an indirect mechanistic pathway involving the γ-Ni[3+]OOH species. The photocurrent and reflectivity measurements show significant changes in the formation of the Ni[3+] oxide when urea is added to the system. The accumulation happens to appear at a higher potential and more slowly.

Electro-ox.: $6\ Ni(OH)_2 + 6\ OH^- \rightarrow 6\ \gamma\text{-}NiOOH + 6\ H_2O + 6\ e^-$ (1)
Chemical ox.: $CO(NH_2)_2 + 6\ \gamma\text{-}NiOOH + H_2O \rightarrow N_2 + CO_2 + 6\ Ni(OH)_2$ (2)

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The Effect of O_2 on the Cathodic Electrochemically Mediated Amine Regeneration Processes via *In Situ* Soft and Hard X-ray Absorption Spectroscopy

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The anthropogenic release of CO_2 is widely recognized as a major contributor to climate change [1], driving the urgent need for effective carbon capture and storage (CCS) technologies. Among different CCS systems, thermal amine scrubbing is widely deployed due to their high CO_2 absorption capacity and fast kinetics [2,3]. Yet, high temperature required for amine regeneration processes (100–140°C), results in significant energy demands and solvent degradation [4]. Electrochemically mediated amine regeneration (EMAR) has emerged as a promising alternative, offering lower energy consumption and reduced solvent degradation due to its operation at ambient temperature and pressure [5]. While several works have been conducted to improve EMAR performance, including reactor designs or amine blend combinations [6,7], the use of EMAR for direct air capture (DAC) applications has yet to be explored. For such applications, not only is there a much lower concentration of CO_2 in the gas, but there is also a higher concentration of O_2 compared to CO_2 rich gas, which may significantly influence the EMAR processes. Addressing this knowledge gap, as part of the Helmholtz Sustainability challenge project DACStorE, the influence of O_2 towards the cathodic EMAR processes were investigated using a combination of electrochemical quartz crystal microbalance (eQCM), *in situ* X-ray absorption spectroscopy (XAS) with soft and hard X-ray, as well as complementary *ex-situ* X-ray photoelectron spectroscopy (XPS) and UV-Vis spectroscopy. Soft XAS at the Cu L_3-edge provided insights into the interface of Cu-electrode and aq. Amine-electrolyte, while hard XAS at the Cu K-edge revealed the nature of Cu species in the bulk aq. amine solution. These experiments indicate the decrease of cathodic EMAR processes' efficiency with increasing O_2 concentration [8]. Smaller effect of O_2 was observed at operation with higher total current densities [8]. Findings from this work provide insight into rational design and optimization of EMAR system for DAC application.

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Hydrogen-Driven Phase Transitions in Palladium Nanoparticles Revealed by *In Situ* BCDI

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The advent of 4th-generation X-ray light sources offers transformative opportunities for *in situ*/*operando* characterization of the structural evolution of nanoparticles in reactive liquid or gas environments. In this presentation, I will illustrate how Bragg coherent X-ray diffraction imaging (BCDI) [1-3] enables direct probing of the strain fields, lattice deformations, and defect dynamics within palladium (Pd) nanocrystals during its hydrogenation. The unique hydrogen (H) absorption properties of Pd underpin its widespread applications in water electrolysis, hydrogenation reaction, hydrogen storage, and hydrogen sensing technologies [4]. However, key questions remain about the mechanisms governing H absorption in Pd crystals, including the nature of the hydride nucleation sites and the dynamics of the H-poor (α) to the H-rich (β) phase transition. Here, we investigated the interplay between H_2 partial pressure and lattice response at room temperature, using *in situ* BCDI at the ID01 beamline of the ESRF. We resolved strain and lattice parameter distributions with nanometer precision during the α-to-β phase transition, providing insights into H absorption dynamics (Figure **1**). We also address open questions on hydride nucleation, including whether it initiates preferentially at structural defects or strained regions, or uniformly across the nanoparticle surface, and whether the α-to-β phase transformation occurs through sharp transitions or two-phase coexistence. This work demonstrates BCDI's ability to visualize structural dynamics in reactive environments, advancing our understanding of phase transformations in Pd systems and paving the way for the optimization of Pd-based materials for use in energy conversion and storage applications.

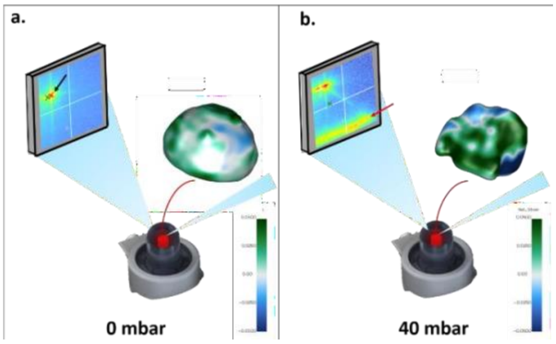

Figure 1: Schematic of the experimental set-up, alongside a reconstructed particle (colored surface strain) under varying H_2 partial pressures. (a) Particle at 0 mbar (vacuum), where only the α phase is present (indicated by the black arrow on the detector). (b) The same particle at 40 mbar, showing the coexistence of the α and β phases (indicated by the red arrow on the detector).

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Insights into the degradation of metallic core-shell nanoparticles under fuel cell conditions by 3D identical location STEM

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Proton exchange membrane fuel cells (PEMFCs) are electrochemical devices capable of generating electricity by oxidizing H2, reformate (H2 rich gas with carbon monoxide (CO) impurities) or other fuels. In recent times, metallic core-shell nanoparticles (NPs) (M@Pt, M=Ru, Rh…) have attracted a big interest as anode catalysts of reformate fed PEMFCs [1]. The high catalytic activity of Pt towards the HOR, together with the CO poisoning tolerance introduced by the accompanying metal make them ideal for heavy-duty applications. Furthermore, since in M@Pt NPs the less stable metal (Rh or Ru) is not directly exposed to the electrolyte, their stability is expected to be higher than in the corresponding alloyed NPs, which commonly suffer from dissolution and dealloying [2]. However, M@Pt can still suffer from degradation under fuel cell conditions by processes that are yet not fully understood, which hinders the design of more stable and durable catalysts.

We investigated the degradation behavior of Rh@Pt NPs by means of identical location-scanning transmission electron microscopy (IL-STEM). This quasi in-situ technique allows to overcome the limitations of the ex-situ techniques, in which only statistical general insights are possible, since in IL-STEM the changes of individual particles are tracked between potential cycles. In particular, we characterized the Rh@Pt NPs after 0, 1000, 4000 and 10000 potential cycles (0.06-0.8V, 0.1V/s). Furthermore, since many of the degradation phenomena take place in 3D (e.g., particle migration and corresponding aggregation), selected regions were reconstructed in 3D by means of electron tomography.

We observed particle migration on the carbon support in all the stages of the potential cycling. However, no widespread particle aggregation was observed, even after 10000 potential cycles. A slight Rh dissolution (up to 5 at.%) during the cycles was detected, which decreased as the number of cycles increased. Even though some small particles dissolved during the first 1000 cycles, the main degradation mechanism responsible for the loss of electrochemically active surface area was found to be particle detachment.

Our results indicate that the investigated Rh@Pt NPs present a remarkable stability, and show how IL-STEM can be used for studying the degradation of catalyst NPs.

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Ionomer-Electrocatalyst Interactions for Fluorine Free Ionomers on Pt Electrodes Using ORR Operando ATR-SEIRAS

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Polyfluoroalkyl substances like Nafion are established and highly investigated materials used as ion conductors. They are used both as membranes and as ionomer coatings in proton exchange membrane fuel cells (PEMFC). Replacing these environmentally persistent materials is a current research focus to make large scale production of PEMFC stacks more environmentally sustainable. Pt-based materials are the state-of-the-art catalysts for anodic and cathodic reactions in PEMFC. Whereas the adsorption on Pt for different functional groups of Nafion (sulfonate and ether groups) has been shown to poison the Pt surface and decrease ORR activity[1], there has been no detailed investigation of these interactions for novel fluorine-free ionomers using surface-enhanced infrared adsorption spectroscopy (SEIRAS). The lack of understanding of the potential-dependent adsorption interactions of fluorine-free ionomers on the Pt-surface limits their meaningful implementation in the next generation of catalyst coated layers (CCLs). In this work, an in-house developed flow-cell adapted to perform operando (attenuated total reflection) ATR-SEIRAS is used. A electrodeposited Pt-layer on the Au enhancement layer is used to investigate the ionomer/catalyst interface interactions for a series of ionomer/monomer described in Figure 1: (a) Nafion, (b) a simplified F-free monomer, (c) a monomer of fluorine-free ionomer coatings and, (d) a fluorine-free ionomer. To assess the impact of binder/catalyst adsorption interactions on ORR activity the ATR-SEIRAS results are correlated to the ORR activity as measured using a rotating disk electrode (RDE) setup.

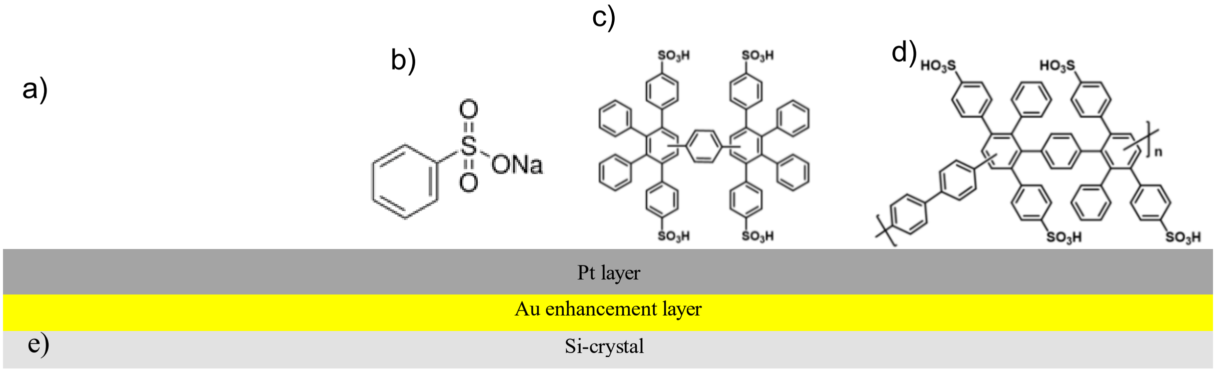

Figure 1: Nafion (a), Sodium benzenesulfonate (b), Sulpho-phenylated dimer (c) and Sulpho-phenylated polyphenylene biphenyl polymer (sPPB) (d) assessed for Pt on Au (e)

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Nanostructured Palladium Electrodes for Reliable Electrochemistry in Liquid-Phase TEM

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Electrochemical devices underpin a wide range of applications, from energy conversion and storage to electrocatalysis and sensing, driving the need for operando techniques capable of capturing electrochemical processes in real time. Miniaturized electrochemical platforms, such as those employed in electrochemical liquid-phase transmission electron microscopy (EC-LPTEM), pose significant challenges that often compromise measurement stability and hinder direct comparison with conventional lab-scale electrochemical data[1]. These limitations arise primarily from the severe geometrical constraints imposed by the EC-LPTEM technique, which impede the implementation of stable and reliable reference and counter electrodes. Conventional reference electrode designs cannot be readily miniaturized, and existing on- chip platforms typically rely on quasi-reference electrodes whose potential stability is degraded under confined conditions, evolving electrolyte composition, and beam-induced radiolysis. Although several strategies have been proposed to mitigate these issues, current solutions often involve complex or bulky configurations[2] or are tailored to specific applications[3], thereby lacking general applicability. Similarly, counter electrodes in microfluidic electrochemical cells frequently suffer from high polarization and parasitic reactions due to their limited electroactive area, preventing them from sustaining the current required by reactions at the working electrode[4]. Here, we report a robust strategy to overcome these limitations by nanostructuring both the reference and counter electrodes using molecular-template-assisted electrodeposition of palladium thin films[5]. This nanostructuring markedly increases the electroactive area while preserving the geometric footprint of the original on-chip electrodes, with the film thickness readily tunable to meet the stringent spatial constraints of EC-LPTEM cells. As a result, the nanostructured counter electrode exhibits strongly reduced polarization during voltammetric cycling of the working electrode, decreasing from more than 3 V for the as-fabricated electrode to approximately 50 mV. In parallel, the stability and reliability of the reference electrode are significantly improved, suppressing potential fluctuations and enabling clearer and more stable cyclic voltammograms and electrochemical measurements. This enhanced stability enables, in principle, direct comparison between operando EC-LPTEM data and conventional lab-scale electrochemical measurements. Overall, these nanostructured electrodes enable reliable electrochemical control in EC- LPTEM setups and, more broadly, in highly confined electrochemical environments, providing a general route toward stable microscale electrochemical measurements under operando conditions.

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Hydrogen evolution reaction on Pt nano-aggregates by correlative multiscale microscopies

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Recent developments focusing in the individual study of nanoparticles during electrocatalysis include scanning probe and scanning electrochemical microscopies. For instance, we have used high throughput optical imaging to monitor the precipitation rate of $M(OH)_2$ due to local pH variations the surface of Pt during HER(*1*). This method allows to quantify the dynamics of the precipitated layer's expansion and uses it as a proxy for the electrocatalytic activity of the NPs. While this methodology that relies on refractive index sensitive optical microscopy (OM) provides high-frequency imaging during the deposition of $M(OH)_2$ at the surface of Pt NPs allowing to track simultaneously the electrocatalytic activity, its spatial resolution is limited. Therefore, in order to assess with higher spatial resolution the electrocatalytically-induced precipitation layer structure, we have developed a multi-scale analysis methodology. Transmission electron microscopy (TEM) in liquid phase, and in particular electrochemical TEM (EC-TEM), represents a direct mean to track structural and morphological changes in direct time and space with nanometric resolution. For instance, great insight in the degradation of electrode and electrolyte materials for batteries has been gained through the studies performed using the electron transparent electrode of EC-TEM

Thus, first, we stablished a protocol to study electrodeposited samples tracked by OM that were later analyze by TEM. Furthermore, we perform in situ EC-TEM observations on such electrodeposited Pt nanoparticles during hydrogen evolution conditions in the presence of transition metal cations similar to OM observations. Here we delineate the extent to which the multiscale approach can be used to correlate information from different microscopy techniques. Moreover, we dive in how while we were able to detect the formation of a layer during electrocatalysis, the electron-beam interaction with the media can have a significant impact on the processes observed and discuss how to deconvolute the information about the electrocatalytic activity from the combined phenomena.

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Investigation Of IrO_2 And Ir Electrocatalysts during OER By In-situ Raman Spectroscopy

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Oxygen evolution reaction (OER) is known to be the limiting water splitting process in proton exchange membrane water electrolyzers (PEMWE). Under acidic conditions, iridium is recognized as one of the most active and the most stable electrocatalyst for OER, which has stimulated the investigation of Ir-based materials using various techniques. Raman spectroscopy can be a powerful tool to gain additional insight into OER mechanism and the active side of iridium, which are still under debate[1,2].

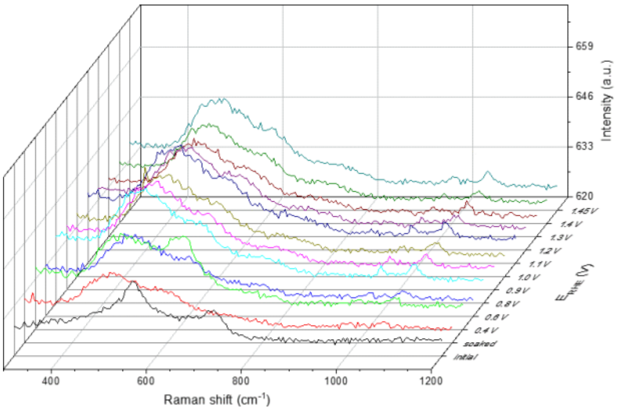

Figure 1: *In situ* Raman spectra of Ir nanoparticles drop-casted on GCE. Electrolyte was 0.1 M $HClO_4$ and spectra were recorded for initial, soaked states, and at potentials from 0.4 to 1.45 V_{RHE}.

This work compares rutile IrO_2 and Ir nanoparticles (Ir NPs) using ex situ and in situ Raman spectroelectrochemistry. Both materials were deposited onto glassy carbon electrodes and electrochemically activated in 0.1 M $HClO_4$. EDS indicates that Ir NPs exhibit partial oxidation prior to activation, consistent with the Raman detection of IrO_2-like features, namely the Eg mode (552 cm^{-1}) and overlapping B_2g/A_1g modes (724 cm^{-1})[3]. IrO_2 and Ir NPs were then activated under three potential protocols (0.05-1.45 V_{RHE}, 0.05-1.60 V_{RHE}, and 1.10-1.60 V_{RHE}), each producing distinct iridium oxidation states and associated OER activities. In situ Raman measurements reveal the emergence of a broad band for Ir NPs during cycling to 1.45 V_{RHE} (figure 1), consistent with previous reports[1,2], whereas rutile IrO_2 largely retains its characteristic Eg and B_2g/A_1g signatures. Ex situ spectra further suggest perchlorate adsorption, with implications for interpretation of in situ Raman assignments. Complementary in situ UV-vis spectroelectrochemistry on FTO-supported samples is expected to provide additional insight into active species and OER mechanisms in both IrO_2 and Ir black systems.

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Combination of Laboratory- and Synchrotron-Based Potentiodynamic Techniques - Opportunities and Challenges

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Synchrotron-based techniques such as soft and hard X-ray absorption spectroscopy provide detailed insight into the atomic and electronic structure of catalytic materials. A drawback of synchrotron experiments, however, is the restricted experimental time. To this end, such experiments are usually thoroughly planned on the basis of previously obtained data in the laboratory. However, the latter are not only a means to an end to prepare the synchrotron experiments but of course contain valuable information on their own. In particular potentiodynamic analysis can identify key potentials for phase transitions as well as dissolution and (re)deposition processes. Importantly, each single type of experiment comes along with specific limitations and/or requirements that must be met. Soft and hard X-ray experiments, for examples, differ significantly in terms of probing depth and sample handling/preparation.[1,2]

Here, some of these technical aspects will be discussed that have to be considered when applying and combining different *operando* techniques performed in the lab as well as at the synchrotron. Further, a detailed example is given how potentiodynamic techniques (soft and hard X-ray absorption spectroscopy, electrochemical quartz microbalance experiments, and Fourier transformed ac voltammetry) were successfully combined to disentangle the dissolution/redeposition processes from changes in the (electronic) structure of a Co based catalyst – stabilized in a lead matrix – for the acidic oxygen evolution reaction.[3]

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From Artefacts to Insights: Challenges and Opportunities in Operando NAP-XPS and XRD of IrOx Catalyst Layers in PEMWE

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Optimizing iridium oxide (IrO_x) for acidic oxygen evolution reaction (OER) requires a deeper understanding of how composition and morphology affect properties.[1] We synthesized unsupported mesoporous IrO_x nanoparticles (2–8 nm) via polyol reduction and calcination (470–670 °C) to examine their distinct sizes and oxidation states relative to a commercial standard.[2] Operando X-ray diffraction (XRD) (0.05–2.00 V) revealed reversible lattice expansion caused by surface reduction Ir^{4+} to Ir^{3+}. Below 0.25 V, the surface becomes partially amorphous due to reduction to metallic Ir^0, with reoxidation observed as slow recrystallization. This structural shifting is directly linked to iridium dissolution, as confirmed by online inductively coupled plasma mass spectrometry (ICP-MS). Long-term membrane electrode assembly (MEA) testing further demonstrated that, initially amorphous, IrOx crystallizes during use and eventually behaves identically to rutile IrO_2. These insights into the redox flexibility of crystalline IrO_2 explain the mechanisms behind catalyst instability during PEMWE start–stop cycling.

Investigations of IrO_x MEAs in a proton-exchange membrane water electrolyzer (PEMWE) cell in near-ambient pressure X-ray photoelectron spectroscopy (NAP-XPS) showed that structural stability and in-plane conductivity of catalyst layers are crucial parameters during operando measurements. Poor conductivity and cracks in the catalyst layer that naturally form during membrane humidification result in poor or lost contact with the current collector, leading to serious artefacts in the measurements. Apart from presenting mitigation strategies for these artefacts, I will discuss ways to exploit them and propose what they can reveal about catalyst layer utilization in a real PEMWE system.

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Posters

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Using Operando Liquid-Electrochemical X-ray and Electron Microscopy and Spectroscopy to Understand Electrocatalytic Processes

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Operando and in situ characterization approaches based on X-ray microscopy (XRM) and transmission electron microscopy (TEM) are increasingly important for resolving chemical and structural evolution in complex electrochemical environments. Unlike post-mortem analysis, operando XRM and TEM enable direct visualization of reactive transformations as they unfold, providing nanoscale maps of chemical state, composition, and morphology during operation. This capability is particularly valuable for catalyst materials, where local oxidation-state changes and phase transitions govern selectivity, stability, and device lifetime. Recent advances in liquid-electrochemical cell hardware have significantly improved the robustness and quantitative fidelity of correlative operando workflows. The incorporation of miniature bulk-scale reference and counter electrodes enables stable electrochemical control and extended cycling durations, supporting time-resolved experiments that capture both transient restructuring and steady-state operation. Together, these developments establish a platform for correlating operating conditions with spatially resolved chemical evolution. For CO_2 electroreduction, operando scanning transmission X-ray microscopy (STXM) is used to map copper oxidation-state dynamics with high sensitivity to cationic Cu species. These measurements reveal phase evolution and restructuring pathways that correlate with changes in catalyst activity and selectivity, including conditions favoring C-C coupling to multi-carbon products. By resolving chemical-state heterogeneity across active regions in real time, operando STXM distinguishes transient and persistent active-state motifs that are otherwise obscured by ensemble-averaged methods. [1] To demonstrate the power of correlative in situ TEM and X-ray absorption spectroscopy (XAS), Cu_2O nanocube restructuring and chemical evolution during nitrate reduction (NO_3RR) to ammonia was also investigated. The nanocubes evolved into distinct structures depending on potential and environment, with Cu_2O stabilized alongside metallic copper under moderately reductive conditions due to surface hydroxide formation, boosting ammonia selectivity at −0.6 V_RHE. Correlating time-resolved spectroscopy with operando microscopy directly links morphological evolution to catalytic mechanisms. [2] Overall, this work demonstrate that operando XRM, TEM, and spectroscopy bridge electrochemical control with nanoscale chemical evolution across electrocatalytic systems, enabling mechanistic insights that inform the design of more selective, durable, and high- performance energy materials.

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Investigating PEMFC Cathode Degradation of Ultra-Small Pt NPs by *Operando* X-ray Scattering

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One of the major challenges for proton exchange membrane fuel cells (PEMFCs) remains the long-term stability. Therefore, understanding the degradation of platinum (Pt) cathode catalysts is crucial for developing more durable PEMFCs. It has been shown that surface oxide formation under oxygen reduction reaction (ORR) conditions is both a crucial step for the catalyst activity, but also for its degradation.[1-3] To mimic this degradation, accelerated stress test (AST) protocols are commonly employed.[4] These dynamic changes in potential cause surface layer oxide-metal phase transitions and subsequent Pt dissolution.

To investigate this degradation behavior under AST protocols two different types of small Pt NPs (1 – 2 nm and 2 – 3 nm diameter) were studied using an *operando* gas diffusion electrode (GDE)[5] and a PEMFC set-up. This unique combination of directly comparing the GDE and PEMFC set-up allows to directly compare the degradation behavior of the same catalyst material. Since amorphous and surface-layer Pt oxides are formed on the particle surface, we studied ultra-small Pt NPs (>3 nm) that exhibit a high surface-to-bulk ratio. To gain insights into the atomic structure of the small Pt NPs that lack long-range order, high energy X-ray total scattering (TS) with pair distribution function (PDF) analysis was employed. In addition, we have combined this with small angle X-ray scattering (SAXS) measurements to give unique insights into the structural evolution of these ultra-small Pt NPs.[6]

With the results from this combined PDF-SAXS study and the comparison between GDE and PEMFC we aim to established the influence of the oxide formation on the degradation behavior of ultra-small Pt NPs, to develop new strategies for mitigating this degradation of PEMFC cathode catalysts.

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How (Not) To Fail An *Operando* Synchrotron Experiment

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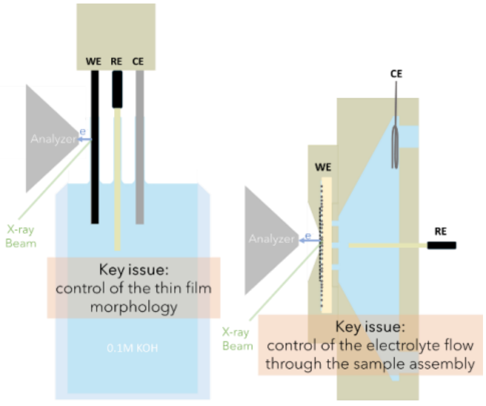
Designing an experimental system for *operando* measurements in a synchrotron is not an easy task, especially when it comes to electrochemistry. Since most phenomena take place at the interface between the catalyst and the electrolyte, the appropriate probing methods should ideally be interface-sensitive. But there is no ideal method. For example, X-ray Photoelectron Spectroscopy (XPS) is a surface-sensitive method that can allow to probe the evolution of the surface composition of a catalyst. However, because of its nanometre-scale probing depth, it requires to "unbury" the electrode-electrolyte interface. This implies very particular electrochemical cell geometries to bring the electrode-electrolyte interface close to the analyzer – and hence, to vacuum – and thus challenging experimental setups.[1,2] On the contrary, X-rays can cross a thick layer of matter, allowing for more user-friendly cell geometries and atmospheric pressure operation for X-ray absorption spectroscopy (XAS). However, XAS spectra are usually dominated by the bulk, which is weakly impacted by electrochemical processes.

Here we focus primarily on *operando* XPS measurements – which are the most challenging – and we discuss the pitfalls that can be faced when preparing a synchrotron experiment. Two geometries are compared: the dip-and-pull cell and the thin window cell. While the first is a very simple design – three parallel electrodes in a beaker – the latter needs a complex sample assembly involving membranes or separators, graphene, and/or silicon nitride windows. But in the end, both methods narrow down to a single crucial issue: how to obtain a catalyst-electrolyte-vacuum interface that is sufficiently stable to perform reliable measurements.



(a) Dip-and-Pull (b) Flow cell

Figure 1 Cell geometries and key features of (a) dip-and-pull and (b) flow cell operando XPS setup

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Disentangling Beam- and Current-Induced Degradation in Operando XAS of Cu Thin Films under CO_2 Reduction Conditions

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Operando X-ray absorption spectroscopy (XAS) is a powerful tool for probing the structural stability of electrocatalysts under realistic reaction conditions. However, at high-brilliance synchrotron beamlines, beam-induced effects can severely interfere with data interpretation, particularly when combined with high electrochemical current densities[1-3]. This challenge is especially critical for operando techniques relying on fixed beam positioning, such as $\Delta\mu$-XANES and fixed-energy XAS, where prolonged irradiation of the same sample region is required[4-6].

Here, we systematically investigate the interplay between X-ray exposure and electrochemical operation during operando Cu K-edge XAS measurements of thin Cu films under CO_2 reduction conditions. Cu thin films supported on PTFE mesh substrates were studied in CO_2-saturated 1 M $KHCO_3$ electrolyte using a custom-designed electrochemical flow cell. To disentangle beam- and electrochemical contributions, four measurement modes were employed, varying X-ray exposure (continuous vs. shuttered) and electrochemical operation (applied cathodic current of -50, to -200 mA cm^{-2} vs. open-circuit potential), while maintaining a fixed beam position throughout the experiment[7].

Unnormalized XAS spectra reveal that continuous irradiation under applied current leads to rapid fluorescence intensity loss and severe local degradation, including substrate perforation and electrolyte leakage at the beam position. In contrast, intermittent exposure substantially mitigates damage, allowing stable operando measurements at high current densities approaching industrially relevant regimes (up to -200 mA cm^{-2}). Control experiments without applied current confirm that X-ray dose alone induces degradation on longer timescales, while electrochemical operation strongly accelerates local failure.

These findings highlight the necessity of beam-damage diagnostics and optimized exposure strategies to ensure reliable operando XAS measurements at modern high-flux synchrotron facilities such as PETRA III.

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Tracking Dynamic Transformations of Cu_2O During Nitrate Reduction via Operando TXM and Multivariate Analysis

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Understanding the dynamic behavior of electrocatalysts under reaction conditions remains a major challenge despite advances in *in situ* and *operando* characterization techniques. A common limitation is the comparison of dynamically acquired data with static references or equilibrium models, even though catalysts often exist in metastable or transient states during operation. These active phases are frequently difficult to detect because their signals are masked by ensemble averaging and the limited temporal resolution of conventional measurements.

Here, we use Transmission X-ray Microscopy (TXM) to correlate morphological and chemical evolution in Cu_2O electrocatalysts during nitrate reduction to ammonia. Using an *in situ* electrochemical cell, we follow the time-resolved transformation of the catalyst under reaction conditions. While conventional linear combination analysis indicates only weak Cu(II) signatures within the spectral background [1], multivariate approaches including Principal Component Analysis (PCA) and Non-negative Matrix Factorization (NMF) enable extraction of chemically meaningful components from hyperspectral datasets.

The analysis reveals transient surface phases forming during catalysis, as well as hydroxide-driven transformations occurring at open circuit potential after the reaction. These results demonstrate that combining TXM hyperspectral imaging with blind source separation provides a powerful strategy to uncover short-lived catalytic states and better resolve the dynamic nature of the solid–liquid interface in electrocatalysis.

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Direct insight into the Pt nanoparticles nucleation and growth by *in situ* electrochemical transmission electron microscopy

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Pt-based metallic nanoparticles play an important role in many heterogeneous electrocatalytic processes. [1-3] Catalytic performance of Pt nanoparticles depends sensitively on particle shape,[2] for instance octahedral Pt nanocrystals facets shows enhanced ORR activity over spherical Pt/C.[4] Therefore shape-controlled synthesis is a powerful lever to tune activity and selectivity for structure-sensitive reactions.[5] Here, we present how the shape of Pt nanoparticles can be tuned by tuning the applied potential during electrodeposition. A first series of Pt particles was produced by pulsed electrodeposition, which yielded spherical-like spiked particles. In a second series of experiments, we synthetized Pt particles by cyclovoltammetry between -1.3V and 1.1V *vs* Ag/AgCl, which yielded cubic nanoparticles. In order to understand how the applied potential influences the nucleation and growth kinetics of Pt particles during CV scans, we performed *in situ* electrochemical cell transmission electron microscopy (*in situ* EC TEM). This setup allows direct visualization of Pt nucleation on a carbon support as a function of the applied potential, allowing to establish a correlation between nanoparticles nucleation and growth and the current-potential curve. *In situ* experiments reveal that Pt NPs nucleate within a narrow potential range around -0.72 /- 0.76 V vs Pt pseudo-reference electrode, and are largely formed during the first CV cycle, leading to relatively dispersed cubic- like and spherical particles (30-70 nm), which is a different morphology from what we observed ex *situ* within comparable potential windows. This result suggests that the conditions used for ex-situ electrodeposition cannot be perfectly reproduced inside the TEM yet, probably due to confined geometry and limited mass transport. In conclusion these results demonstrate that the shape of Pt nanoparticles can be tuned by playing with the applied potential (pulsed ED or CV). *In situ* STEM is a potentially powerful technique to establish a link between the nucleation and growth kinetics, the nanoparticles shape and applied potential, however, some key point such as the effect of the electron dose and system confinement on growth mechanism must be studied in more details to validate the *in situ* S/TEM approach.

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Unravelling the Blessing and Curse of IrCu Aerogel Catalyst for the Oxygen Evolution Reaction

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Proton exchange membrane water electrolyser (PEMWE) technology is rapidly expanding to meet growing hydrogen demand and support industrial decarbonization [1]. However, its large-scale deployment is constrained by the limited global availability of iridium (Ir), which is required to catalyze the limiting oxygen evolution reaction (OER) at the anode [2]. While increasing Ir utilization by supporting nanoparticulate Ir on doped metal oxides has shown promising results, the issue translates to the stability of the support [3]. An alternative approach is to use a nanostructured, unsupported Ir-based catalyst aerogel which combines both high intrinsic activity and high specific surface area. Herein, we successfully synthesised an iridium-copper (IrCu) aerogel originally proposed by Shi et al [4]. Its desirable electrocatalytic activity towards OER compared to benchmark catalysts was confirmed in both liquid electrolyte and single-cell PEMWE measurements. The reasons for such activity enhancement were investigated using a broad portfolio of ex-situ and operando characterization techniques. Notably, operando wide angle X-ray (total) scattering (WAXS) allowed tracking of the microstructure (crystallite size, lattice strain and microstrain) of the IrCu aerogel in comparison to a house-made carbon-supported IrCu/C academic benchmark catalyst during electrochemical operation. The results show that the high OER activity of the IrCu aerogel originates primarily from the presence of lattice distortions, i.e., through a structural effect rather than an alloying effect. Moreover, beyond decisively influencing the electrocatalytic activity, pair distribution function (PDF) analysis of the WAXS patterns (Figure 1) demonstrates that the aerogel structure inhibits the formation of stable Ir oxide species during operation, unlike for isolated nanoparticles, which is detrimental to the catalyst stability [5].

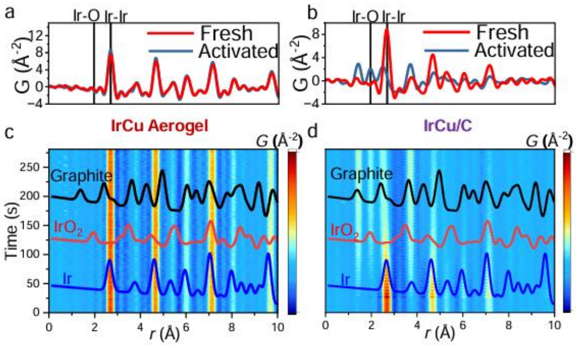

Figure 1. *In situ* pair distribution function (PDF) G(r) patterns collected before (fresh) and after activation for a) IrCu aerogel and b) IrCu/C. c-d) *In situ* PDF G(r) waterfall plots for c) IrCu aerogel and d) IrCu/C over time, with calculated PDFs of graphite (black), rutile IrO_2 (red), and metallic Ir (blue) overlaid as references.

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Investigation of Nickel-Based Electrocatalysts for Hydrogen Oxidation Reaction with Operando X-ray Scattering

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The energy transition demands sustainable solutions, with hydrogen fuel cells and electrolysers playing a critical role in enabling energy storage, conversion, and utilization. A key challenge lies in developing electrocatalysts that combine high activity, stability, and affordability. Nickel-based catalysts have emerged as promising alternatives to costly platinum group metals (PGMs), especially in alkaline environments. However, the interplay between their structure, properties, and catalytic activity—particularly the role of nickel oxidation—remains elusive, necessitating further studies to better understand the existing materials and further optimise their performance[1].

Advanced operando X-ray techniques offer a pathway to understand these complexities. By providing real-time insights into structural and chemical transformations, these methods enable a deeper understanding of the catalytic behaviour[2].

Here, operando X-ray total scattering combined with atomic pair distribution function (PDF) analysis has been employed to monitor the oxidation trends of core@shell Ni@C catalysts during the hydrogen oxidation reaction (HOR) in aqueous KOH electrolyte, simulating the environment of anion-exchange membrane fuel cells (AEMFC). This poster will present recent results obtained with operando X-ray techniques applied to Ni@C materials developed by our group and derived from a Ni-based metal organic framework, which has shown state-of-the-art performance in lab-scale AEMFC.

The findings improve our understanding of the material's behaviour regarding reversible and irreversible Ni oxidation stages and will support the design of more efficient Ni-based catalysts as well as protocols to activate or reactivate them in an AEMFC.

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Influence Of Metal Centre on Metal-Phthalocyanine Redox Potentials

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The applicability of homogeneous electrocatalysts is often constrained by accessible redox potential windows, which govern overpotential, and catalyst stability under operating conditions. Metal phthalocyanines are attractive single atom catalysts because both the macrocycle and the central metal can, in principle, be modified to tune redox properties. However, the relative influence of the metal atom versus the ligand on metal phthalocyanine redox potentials remains unclear. Resolving this issue should bring greater clarity to design strategies for conjugated metal-ligand catalysts.

In this work, we use density functional theory to systematically evaluate the redox potentials across a broad series of metal phthalocyanines spanning 3d transitions metals, as well asCa, Mg, Al, and In, and overall charge states of +1 to -4. Redox potentials are referenced to an effective absolute potential based on acetic acid enabling comparison across solvents such as water, acetonitrile and tetrahydrofuran. Our preliminary results indicate that the effect of the metal atom depends on the redox couple. The maximum variation in redox potentials is smallest for the -1/-2 and -2/-3 redox couples and increases as the system is more oxidized or reduced. Orbital analysis shows the HOMO and LUMO orbitals mostly localized on the phthalocyanine which supports ligand-centred redox chemistry across the set. Electrocatalytic reactions such as the HER and the CO2RR occur over the potential window that encompasses the -1/-2 and -2/-3 redox couples; therefore, performance differences are more likely due to metal-intermediate interactions. In contrast, for reactions requiring more strongly oxidizing or reducing potentials, the metal's identity will be of greater significance to the catalyst's stability.

In Situ Spectroscopic Studies of Electroinduced and Catalytically Reversible Reactions of Molecular Uranium Complexes

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Interconversion of actinide oxidation states is crucial in the fuel cycle for low-carbon nuclear power and contributes to nuclear technology development. The redox behaviors of uranium species govern the viability of most processes in this realm, but use of electrochemical methods to drive reactions of molecular uranium complexes and to obtain molecular insights into the outcomes of electrode-driven actinide reactions has received far less attention than it deserves. In this poster, two reversible and electrocatalytic systems for stepwise functionalization of the typically unreactive oxo groups of the uranyl dication (UO_2^{2+}) with exogenous triphenylborane (BPh_3) serving as a moderate electrophile will be discussed.[1] The results of parallel chemical, electroanalytical, and structural data from X-ray diffraction analysis on key reduced and borylated products demonstrate that our electrochemical approach largely avoids undesired cross reactions[2] and disproportionation pathways; these usually impact the multicomponent systems needed for uranyl functionalization chemistry. Our recent work focuses on comparisons of *ex situ* bulk electrolysis studies and *in situ* spectroelectrochemical investigations with UV-visible-NIR absorption spectroscopy, and has confirmed that the in situ methods provide a reliable, near real-time view of the actinide redox interconversions happening in solution. Building on these findings, *in situ* measurements are currently underway in which advanced X-ray spectroscopies, including M_4-edge XANES and RIXS, are being pursued to obtain insights into bonding properties of reactive intermediates. Taken together, the results show that electrochemical and *in situ* spectroelectrochemical methods more commonly employed in renewable energy and fuel conversion schemes can also be quite usefully employed for selective interconversion of molecular actinide species, reminiscent of methods commonly employed in transition metal redox catalysis.[3,4]

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Investigating Nickel Nanoparticles During Hydrogen Evolution Reaction by In-situ Electrochemical Transmission Electron Microscopy

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Nickel nanoparticles (Ni NPs) are promising non-noble catalysts for the hydrogen evolution reaction (HER), especially in alkaline electrolytes, because they are active, abundant, and low-cost. Their nanoscale dimensions provide a high density of tunable active sites, tunable surface facets, and enhanced metal-electrolyte interactions. However, under HER conditions, the structure and surface chemistry of these NPs can dynamically evolve in response to applied potential, local chemistry, and interaction with adsorbed intermediates, which can drive surface oxidation/reduction, formation of $Ni(OH)_x$ species, facet/defect restructuring, and in some cases dissolution-redeposition. These changes can either improve or degrade performance by altering active-site availability and reaction kinetics, making it essential to track Ni NP structure and chemistry under operating conditions. Direct visualization at high spatial resolution can provide invaluable insights into these processes. Recently developed electrochemical liquid-cell transmission electron microscopy (EC-TEM) holders enable in-situ nanoscale study of material reactions under bias[1].

In this work, we first studied the electrochemical deposition of Ni NPs in-situ at high resolutions, and then we investigated the morphological/structural/chemical modifications of these NPs in HER conditions in an alkaline solution. The in-situ EC-TEM experiments were performed using a Protochips Poseidon 500 liquid holder at a JEOL JEM ARM-200 CF using a 200 kV accelerating voltage in the bright-field mode. Experimental images and videos were recorded under standard illumination conditions for real-time imaging, with controlled electron-beam damage. The electrodeposition process of Ni NPs was conducted from 0.2 M $(NH_4)_2SO_4$ and 0.02 M $NiSO_4$ solution in ultrapure deionized water with both chronoamperometric and chronopotentiometric processes. Circular-shaped NPs ranging between 10 and 100 nm were grown on the electron-transparent glassy-carbon electrode. These NPs were then subjected to the HER process in a Na_2CO_3-based alkaline solution. The morphological and structural/chemical modifications were studied by collecting the images and diffraction patterns, respectively, during the applied bias. The results provided invaluable insights into the restructuring of electrocatalytic particles during reactions.

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Novel Operando-XMCD Approach To Study Catalyst Evolution During OER On The Example Of Nickel Electrode

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OPERANDO-XMCD APPROACH

The transition to green energy relies heavily on efficient energy storage and utilization, with hydrogen offering a sustainable solution for It. While water electrolysis offers a sustainable route to hydrogen generation, the oxygen evolution reaction (OER) remains a kinetic bottleneck due to its sluggish kinetics. Recently, it has been reported that using spin ordered materials as catalysts under magnetic fields allows to increase the OER catalytic efficiency[1,2], which in turn promises a more energy-efficient green hydrogen economy. The exact nature of this enhancement, however, still remains to be uncovered and more advanced techniques are required to fully understand the correlation between magnetic properties and OER activity under operating conditions[3].

We have employed a well-established technique of X-ray Magnetic Circular Dichroism (XMCD) spectroscopy and adapted it to be performed operando under electrochemical conditions. 5 nm Ni (working catalyst) and 3 nm of Ti (adhesion layer) were sputtered on a Si_3N_4 X-Ray transparent membrane and put into the custom designed vacuum-tight operando electrochemical cell with 2 permanent magnets generating an in-plane field of approximately 0.1 T. Thus, XMCD spectra collection was possible by switching between circular-left and circular-right polarizations in the grazing incidence geometry.

Throughout the experiment we have studied XMCD properties of Ni catalyst under several conditions, including in vacuum, at open circuit conditions and during OER and HER. We have successfully tracked how the oxidation state of the nickel layer changes depending on the surrounding conditions and applied potential, while simultaneously tracking magnetic property evolution. We have found that despite going to oxidizing OER potentials, there was still evidence of metallic nickel present as evident not only by its X-ray absorption spectroscopy (XAS) fingerprint, but also by the unique XMCD shape. Our findings demonstrate the potential of our newly developed operando-XMCD approach to achieve new insights into the nature of magnetically enhanced electrocatalysis, and we provide an experimental guideline to perform such measurements.

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Challenges Of Attenuated Total Reflection Surface-Enhanced Infrared Absorption Spectroscopy (ATR-SEIRAS) For Powder Catalyst Studies

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Attenuated Total Reflection Surface-Enhanced Infrared Absorption Spectroscopy (ATR-SEIRAS) coupled with electrochemistry has remained a popular tool for reaction mechanism investigation for the past 25 years. Developed by Osawa (1-3) in 1980-1990s, the method has mainly been applied to metal films formed on the surface of an internal reflection element (4-6). In such configuration, the infrared signal is enhanced thanks to the Surface-Enhanced Infrared Absorption (*SEIRA)* effect emerging in the immediate vicinity of metal film, thus allowing to observe infrared (IR) vibration bands of the species adsorbed on the metal film surface. In 2007, Watanabe (7) introduced a modified method where a catalyst of interest (e.g., Pt/C) was deposited onto a gold film, however, the method being named *"ATR-IR"*. Nevertheless, the method spread among the scientific community under the name *"ATR-SEIRAS"* (8-13). According to the literature, the bands of species adsorbed on the catalyst particles that are close to the gold film are enhanced by SEIRA effect induced by the gold film (10,13).

However, such configuration is very complex and the phenomena at play are not truly established. It is known that SEIRA effect is short-ranged (< 10 nm), that the enhancement is non-uniform across the surface and depends on IR wavelength, metal film particles size and shape, and on the incident angle of the IR beam (3,14). Moreover, in ATR configuration total internal reflection results in an electromagnetic field at the prism / electrolyte interface, called "*evanescent wave",* which extends from the prism surface into the electrolyte over several hundred nanometres, and it may also contribute to the recorded spectra. In this work, we investigate the system comprised of a Si ATR element with and without gold film deposited on top, and Pt/C catalyst, and try to deconvolute the roles of the evanescent wave and SEIRA effect.

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Atomic-Level Insight Into Multi-Carbon Product Formation Via CO2RR On Chiral Catalysts

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The global transition from fossil fuels to renewable energy is pivotal for combating climate change, with electrochemical CO_2 reduction (CO_2RR) emerging as a promising strategy for carbon mitigation. By coupling renewable electricity with efficient CO_2 utilization, CO_2RR enables the direct conversion of carbon dioxide into value-added chemicals and fuels. However, the selective generation of complex multi-carbon products remains a formidable challenge, rooted in the intricate interplay between catalyst structure, composition, and reactivity [1,2,3].

Recent breakthroughs such as harnessing spin polarization effects via external magnetic fields or chiral molecular environments have demonstrated remarkable enhancements in catalytic performance, particularly for copper-based systems [4,5,6].

In this research, we focus on the design and investigation of chiral CuO_x nanostructures, which uniquely combine the catalytic versatility of copper with the tunable properties of oxide interfaces. Cu is nearly the only metallic catalyst capable of promoting C–C coupling through CO* dimerization, making it an ideal platform for exploring spin-dependent effects and selective CO_2RR.

Advanced electron microscopy techniques, especially (Scanning) Transmission Electron Microscopy ((S)TEM), are employed to unravel the atomic-scale features of these chiral CuO_x catalysts. Correlative (S)TEM (performed before and after reaction) enable visualization of active sites and tracking of structural evolution. These insights are crucial for understanding degradation mechanisms, optimizing catalyst design, and ultimately improving selectivity and efficiency.

By integrating state-of-the-art electron microscopy with the synthesis of robust, intrinsically chiral CuO_x electrodes, this research paves the way for the rational design of next-generation electrocatalysts.

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Unveiling Solid-Liquid Interface Interactions through X-ray Absorption Spectroscopy

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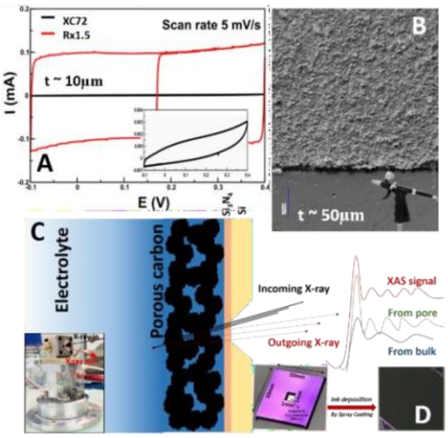
Electrochemical devices—such as metal-ion batteries, supercapacitors, and electrolyzers—are key technologies in energy conversion and storage systems, with their performance largely dictated by processes occurring at the **surface–electrolyte interface**. The local structure of this interface governs both the thermodynamics and kinetics of electron transfer in batteries and electrolyzers, while also controlling charge accumulation and release in supercapacitors. A detailed understanding of this **interfacial structure** is therefore essential to optimizing electrochemical performance.[1] In this respect, we have focused specifically on **porous carbon materials** for their huge electrochemical surface area (ECSA) as they can serve as fundamental models for the behavior of electrolyte at the solid-liquid interface (through the numerous pores) providing a dominating signal over the bulk. The specific porous carbon material, called **Rx1.5**, studied here features pores of ***1.1 nm*** in diameter and a surface area of ca. ***1740 m²/g***.[2] As expected, the electrochemical performances of this material show a hundred-fold increase in capacitance compared to Vulcan XC72, as shown from the current intensity measured by cyclic voltammetry (fig. 1A). To elucidate the structure and spatial organization of electrolyte ions (K^+, Na^+ and Cl^-) at porous carbon electrode surfaces under operating electrochemical conditions, we designed a specialized electrode (Au-coated Si_3N_4/SiO_2) optimized through careful control of **ink formulation and deposition methods** (fig. 1B, D), that has been studied for **synchrotron-based in-situ XANES** measurements[3,4] using a vacuum-compatible electrochemical cell.[5] The signatures of the XANES spectra: **edge position, intensity, and pre-edge features** acquired from the bulk electrolyte and confined electrolyte (in the pores of electrode) yield information on the interaction of these ions with the carbon surface and their structure at the solid–liquid interface. The strategy of qualitative XANES analysis involves the electrode-electrolyte signal comparing against, and deconvoluted from, bulk electrolyte spectra (fig. 1C). Furthermore, the obtained data will be confronted by theoretical calculations for deeper understanding of solid-liquid interface interaction.



(A) Cyclic voltamograms of Rx1.5 vs XC72 electrode of thickness 10 µm in 0.3 M KCl. (B) Optical image of electrode, prepared by Rx1.5 ink deposition by spray coating (thickness t ~ 50 µm). (C) Strategy of ongoing XANES data analysis deconvoluting bulk vs pore confined electrolyte signals (inset: electrochemical cell for synchrotron measurement). (D) Electrode preparation by ink deposition on Au-coated Si_3N_4 membrane (200 nm).

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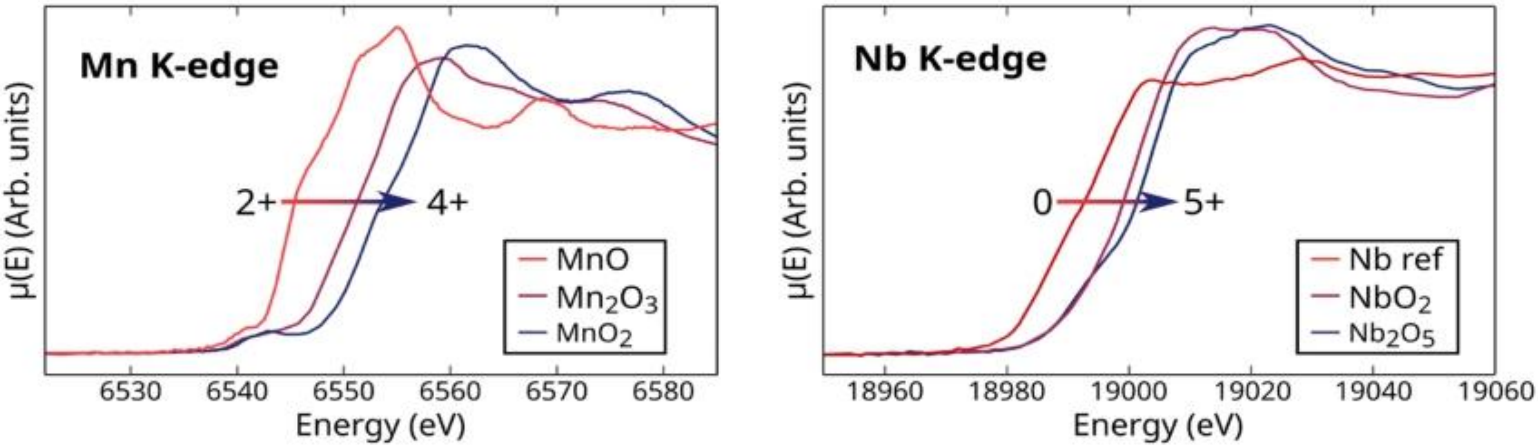
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4+
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Energy (eV)
Nb K-edge
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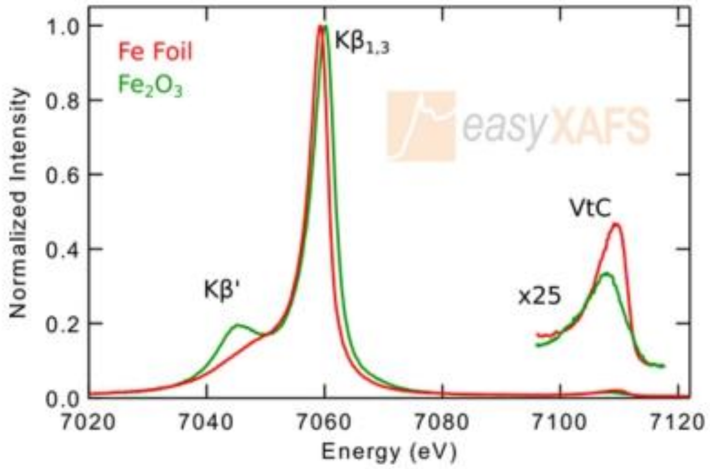
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Energy (eV)

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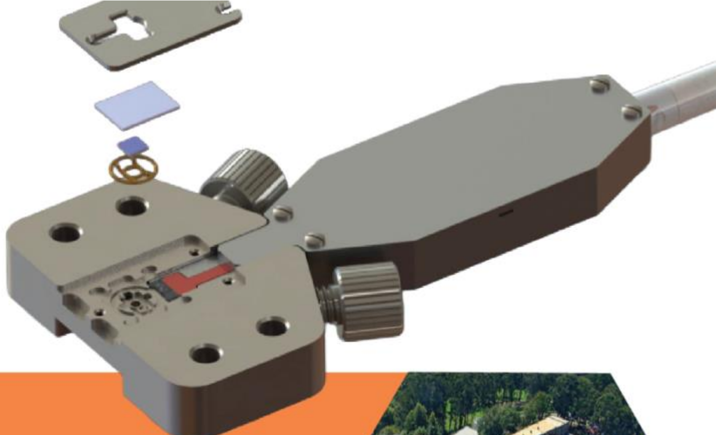

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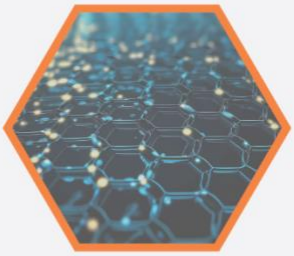

NANOMATERIAL SYNTHESIS

CATALYSIS

Operando Resonant Soft X-ray Scattering Studies of Chemical Environment and Interparticle Dynamics of Cu Nanocatalysts for CO2 Electroreduction

Yao Yang, Inwhan Roh, Sheena Louisia, Chubai Chen, Jianbo Jin, Sunmoon Yu, Miquel B. Salmeron, Cheng Wang, and Peidong Yang

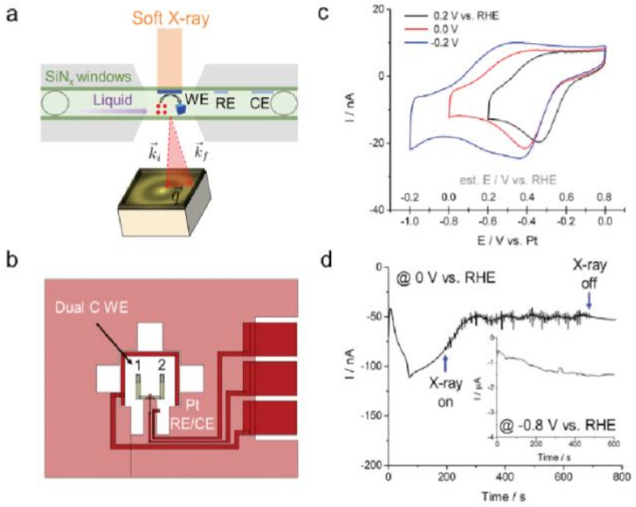

Figure 1. Operando electrochemical liquid-cell resonant soft X-ray scattering (EC-RSoXS) setup. (a) Schematic of the X-ray cell with the capability to enable reliable electrochemistry and simultaneously acquire soft XAS and SAXS through 1 μm liquid. (b) Design of the X-ray microchip with dual carbon WE, and Pt RE and CE. (c) CV profiles of Cu NPs at 100 mV/s in CO2-saturated 0.1 M KHCO3 with different lower potential limits. (d) CA profiles of Cu NPs at 0 and −0.8 V (inset) vs RHE

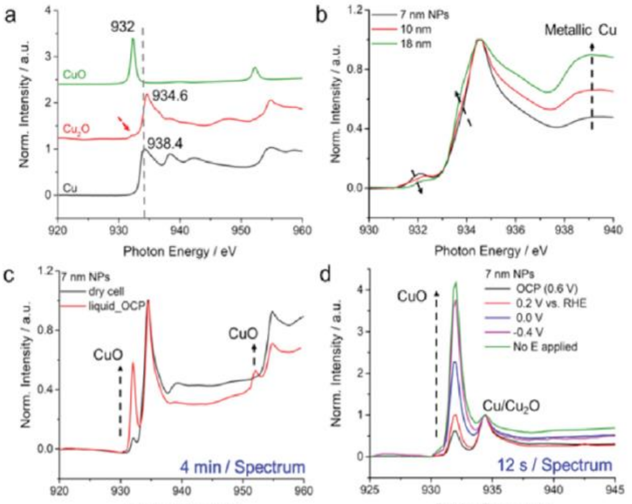

Figure 2. Operando soft XAS of the 7 nm NP ensemble in electrolyte under X-ray exposure. (a) XAS spectra of standard references and (b) 7, 10, and 18 nm NP ensembles. (c) XAS spectra of the 7 nm NP ensemble showing beam-induced oxidation to CuO in liquid. (d) Operando XAS spectra of the 7 nm NP ensemble under applied potentials from 0.2 to −0.4 V vs RHE, showing that X-ray-induced oxidation overwhelmed electroreduction.

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