# Probing Catalysts in Action: Multi-technique Operando Studies of Nanoparticle Electrocatalysts

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Electrocatalysts are central to key energy conversion technologies such as fuel cells and water electrolyzers. Often, scarce and precious metals are needed for efficient catalysis, so nano-sizing materials is crucial to maximize the number of active sites. However, the structure of nanoparticle electrocatalysts is highly dynamic under the harsh operating conditions applied. Understanding how catalyst nanoparticles evolve during electrochemical reactions is therefore essential for linking structure, stability, and catalytic performance. I will present recent work on multi-technique in situ and operando characterization of nanoparticle electrocatalysts, with high-energy X-ray scattering methods.[1]

By combining X-ray powder diffraction, small-angle X-ray scattering, and total scattering with pair distribution function (PDF) analysis, we can probe structural changes in nanoparticles as they actively drive electrochemical reactions. This multiscale scattering approach enables simultaneous insight into crystallographic structure, local order, and nanoparticle size and morphology, allowing us to follow catalyst activation, restructuring, and degradation in real time. For this purpose, we have designed a dedicated operando cell for high-quality scattering measurements at synchrotron facilities.[2] Complementing scattering data with X-ray absorption spectroscopy provides additional insights into oxidation state and element-specific local coordination.[3] Examples will include *operando* studies of iridium-based oxygen evolution catalysts, where total scattering reveals the formation of highly disordered oxide domains under reaction conditions, as well as investigations of Pt-based nanoparticles relevant for fuel cell catalysis.[3,4] In addition, I will highlight work tracking the formation and evolution of compositionally complex nanomaterials, illustrating how in situ and operando X-ray techniques can elucidate both synthesis pathways and catalytic behaviour.[5]

Combining complementary high-energy X-ray methods provides a powerful framework for understanding functional nanomaterials probed under realistic operating conditions. This operando multi-technique approach offers new opportunities toward direct observation of catalysts in action, ultimately enabling more rational design of stable and efficient electrocatalysts for energy applications.

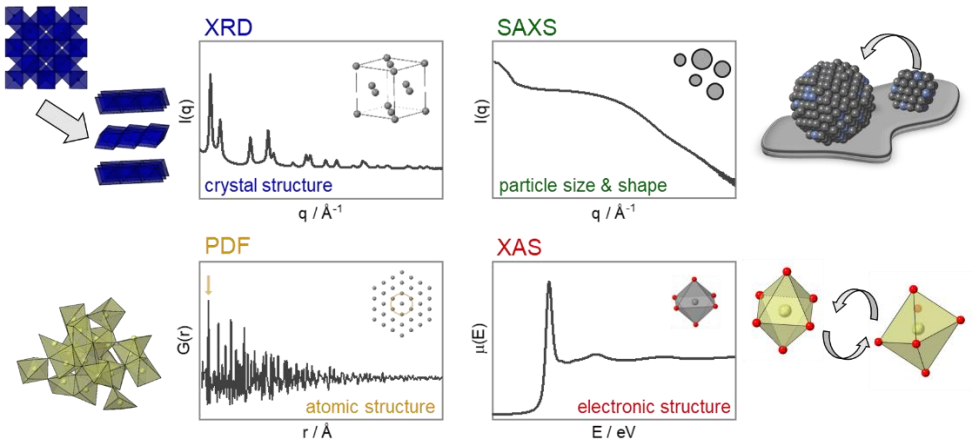



***Figure 1.*** *Toolbox of X-ray techniques used for the operando characterization of electrocatalysts for energy conversion reactions, with corresponding relevant restructuring mechanisms that can be probed.[1]*

[1] R.K. Pittkowski, ChemElectroChem. (2024). e202400171
[2] G.K.H. Wiberg, R.K.Pittkowski et al. Chimia. (2024). 78, 344-348.
[3] R. K. Pittkowski et al. J. Am. Chem. Soc. (2024). 146,27517-27527.
[4] J. Schroeder, R. K. Pittkowski et al. ACS Catalysis (2022). 12, 2077-2085.
[5] R. Pittkowski et al. EES Catalysis (2023). 1, 950-960.