Elucidating Reaction Kinetics of oxide-derived copper electrocatalysts for CO_2 reduction with In-Situ Small- and Wide-Angle X-ray Scattering Experiments

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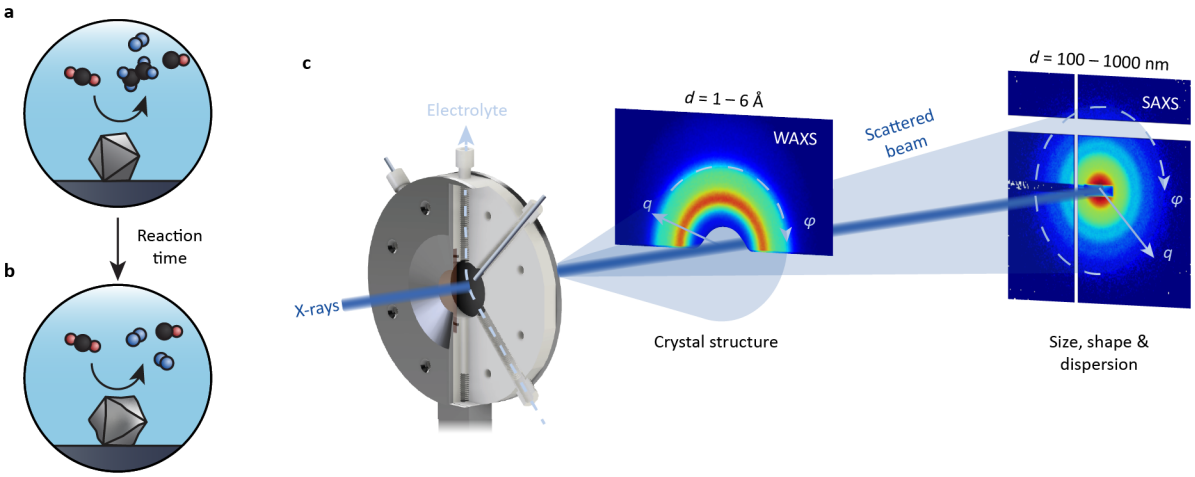
Nanocrystals have a highly dynamic structure, constantly reorganizing both during synthesis and during use in applications. Structural transformations can enhance or degrade their properties. Understanding the transformation kinetics is key in steering the nanocrystals towards a structure with desired properties. Tracking structural changes with real-space characterization faces limitations, such as low statistics in electron microscopy or poor spatial resolution in light microscopy. X-ray scattering techniques offer ensemble-scale statistics across length scales from the interatomic to the interparticle, within a single experiment and with high temporal resolution.

In this presentation I will discuss how we used in-situ X-ray scattering experiments to monitor the structural transformations of octahedral copper electrocatalyst particles. I will discuss the activation and deactivation of oxide-derived particles during CO_2 reduction and the effect of the structural changes on the catalytic performance.[1]



(a-b) Cartoons showing the main products formed during electrochemical CO_2 reduction over octahedral Cu_2O electrocatalyst particles at **(a)** early stages of cathodic bias and **(b)** after prolonged operation up to 30 min. Over the course of the reaction catalytic selectivity and activity is reduced. **(c)** Illustration of electrochemical cell used for the in situ X-ray scattering experiments. Detectors show representative 2D in situ wide- and small-angle X-ray scattering patterns and the length scales probed with wide-angle X-ray scattering and small-angle X-ray scattering. The dashed arrows show the azimuthal integration of the data and the solid arrows the scattering vector *q*.

[1] J. de Ruiter, V. R. M. Benning, S. Yang, B. J. den Hartigh, H. Wang, P. T. Prins, J. M. Dorresteijn, J. C. L. Janssens, G. Manna, A. V. Petukhov, B. M. Weckhuysen, F. T. Rabouw & W. van der Stam, *Nat. Commun.*, **2025**, *16*, 373