ESRF	**Experiment title:** Tracking structural response of Cu-based electrocatalysts during potentiodynamic CO_2 electroreduction using in situ time-resolved high energy X-ray diffraction and scattering	**Experiment number**: CH5914
Beamline: ID31	**Date of experiment**: from: 08 Sep 2021 to: 14 Sep 2021	**Date of report**: 03 Mar 2022
Shifts: 18	**Local contact(s)**: Jakub Drnec	*Received at ESRF:*

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Report:

The goal of this experiment was to identify the dynamics of the (bulk) structural and morphological response of oxide-derived Cu and Ag-doped Cu electrocatalysts to potentiodynamic electrochemical and CO_2 electroreduction (CO_2RR) conditions. Therefore, we aimed to record high energy diffraction pattern with ms time resolution during potential pulse experiments for varying potential ranges. Pulsing between and within capacitive, Faradaic, and catalytic conditions in CO_2-containing and CO_2-free electrolytes will allow us to differentiate between the bulk lattice response to the charged state, the surface chemistry and active state of the catalysts. We also planned previously to comparatively study size-selected Ag and Au particles as well well as complementary operando small-angle X-ray scattering measurements.
We successfully performed operando HE-XRD experiments on a variety of decorated Cu_2O nanocubes (NC) during electrochemical CO_2 reduction reaction (CO_2RR) under potentiostatic and potentiodynamic reaction conditions. Therein, we could follow the electrochemical reduction of the Cu_2O decorated with Ag, Au and ZnO under the reducing conditions by recording diffraction pattern during constant CO_2RR potential and for selected increasingly cathodic electrode potentials. We could follow the structural interaction of the Au with the oxide-derived Cu as well as subtle reversible changes in the amplitude of the ZnO diffraction feature under reducing and oxidizing conditions. We performed various potentiodynamic experiments e.g. under pulsed CO_2RR conditions as well as cyclic voltammetry. We are currently working on the detailed data analysis primarily using Rietveld refinement.
In addition to the *operando* experiments on the shape-selected Cu_2O, we performed preliminary experiments on single-atom catalysts for CO_2RR. We did not perform any SAXS experiment to more efficiently use the time for the diffraction experiments. The planned experiments on the pure Cu_2O nanocubes as well as most of the experiments on the Ag-Cu_2O we could already perform in another beamtime.