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Evaluation of the impact of the in situ cell design on the interfacial speciation over C-based electrodes during room temperature CO2 electroreduction

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Electrochemically or photo-electrochemically activated, hydrogenation and dehydrogenation reactions of small molecules (e. g. H2O, CO2, O2, N2) are at the core of currently explored decarbonization technologies. These technologies are key to delivering net zero emissions. The major challenges to face for boosting innovation in this area are common to these reactions: reducing material costs; improving the energy efficiencies to practical levels and reducing material degradation during operation. The understanding of the underlying mechanisms, including restructuring and compositional change of the electrode surface upon polarization, activation, and deactivation is acknowledged to guide towards the synthesis of improved materials. This knowledge can be obtained with highest resolution of chemical states via surface-sensitive in situ spectroscopic techniques such as ambient pressure X-ray photoelectron and absorption spectroscopies that investigate the electronic structure at surfaces and interfaces. However, there are technical challenges to overcome for the realization of electron detection-based in situ studies of liquid/solid electrified interfaces: the inelastic mean free path of electron and the high absorption cross section in the soft X-ray regime limit the analysis to thin liquid films wetting the electrode surface.
In the last decade progress in this field has been gigantic and different approaches have been proposed, amongst which the suitability of electron-transparent graphene membrane to separate the liquid environment from the vacuum chamber has been the focus of recent investigations carried out in collaboration with the beamline research team at the ISISS beamline of the synchrotron facility BESSY II at Berlin.

In this contribution, I will focus on PEM-based systems to study the electrocatalytic conversion of CO2 to valuable products over carbon-based electrodes. I will describe the technical aspects of the various cell designs used, spanning different pressure ranges, from a humidified gas/solid interface [1] to a stagnant liquid film/solid interface [2]. I will compare the surface dynamics observed for chemical equivalent electrode systems in the different cell configurations, including the results obtained using a fluorescence yield based continuous flow cell. [3]

Supporting literature:
[1] V. Pfeifer, T. E. Jones, J. J. Velasco Vélez, R. Arrigo, S. Piccinin, M. Hävecker, A. Knop-Gericke, R. Schlögl, In situ observation of reactive oxygen species forming on oxygen-evolving iridium surfaces, Chem. Sci., 2017, DOI: 10.1039/C6SC04622C.
[2] J. Velasco-Vélez, E. Carbonio, C.-H. Chuang, C.-J. Hsu, J.-F. Lee, R. Arrigo, M. Hävecker, R. Wang, M. Plodinec, A. Centeno, A. Zurutuza, L. Falling, R. Mom, S. Hofmann, R. Schlögl, A. Knop-Gericke, T. Jones, Surface constrained electron-hole rich species active in the electrocatalytic water splitting, https://orcid.org/0000-0002-6595-0168.
[3] J.-J. Velasco-Vélez, C.-H. Chuang, D. Gao, Q. Zhu, D. Ivanov, H. Sang Jeon, R. Arrigo, R. V. Mom, E. Stotz, H.-L. Wu, T. E. Jones, B. Roldan Cuenya, A. Knop-Gericke, R. Schlögl, On the Activity/Selectivity and Phase Stability of Thermally Grown Copper Oxides during the Electrocatalytic Reduction of CO2, ACS Catalysis 2020 10 (19), 11510-11518.

Presenter: Dr ARRIGO, Rosa