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Interface Sensitive Drain Current X-ray Absorption Measurements of Operando Electrochemical Cells

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Electrochemical processes depend on several phenomena, such as water-ion interactions, diffusion, adsorption and the chemical state of the electrode surface. These processes occur in a thin layer at the electrolyte/electrode interface, the electrical double layer (EDL). The required interface sensitivity to investigate the EDL is challenging to achieve due to a lack of techniques that are compatible with operando electrochemical cells. In fact, the commonly used fluorescence yield mode of operando X-ray absorption spectroscopy (XAS) is considered as a bulk sensitive measurement. Here we describe a novel detection technique for XAS by using the drain current of the electrochemical working electrode to achieve the required interface sensitivity. Usually, the XAS drain current is buried below the several orders of magnitude higher electrochemical current and cannot be accessed. To overcome this issue, the X-ray beam was amplitude modulated by a mechanical chopper that was implemented in the UE56/2-PGM1 beamline at BESSY II. We have developed advanced separation electronics that are capable of separation the modulated drain current (AC) from continuous electrochemical current (DC). The new electronics provide a high dynamic range for the AC signal, efficient filtering of noise introduced by the potentiostat, and they are transparent for the operation of the electrochemical cell. The capabilities of this approach were shown by in-situ electrodeposition of copper using the detection of O K-edge and Cu L-edges. This technique enables the investigation of the surface structure of the deposited Cu, the concentration of the dissolved Cu ions in the EDL and the interfacial water structure. We found that the deposition occurs via Cu2O/CuOH intermediates followed by a reduction to metallic copper rather than a direct process. This result highlights the complexity of interfacial electrochemistry, and the need to resolve it in molecular-level detail.

I plan to submit also conference proceedings

No

Primary author: Dr VELASCO VÉLEZ, Juan J. (ALBA Synchrotron Light Source)

Co-authors: Dr GROSSE, Nicolai B. (Fritz-Haber-Institut der Max-Planck-Gesellschaft); ZELLER, Patrick (Fritz-Haber-Institut der Max-Planck-Gesellschaft); Dr KNOP-GERICKE, Axel (Department of Heterogeneous Reactions, Max Planck Institute for Chemical Energy Conversion); Prof. SCHLÖGL, Robert (Fritz-Haber-Institut der Max-Planck-Gesellschaft); Prof. MOM, Rik V. (Leiden Institute of Chemistry)

Presenter: ZELLER, Patrick (Fritz-Haber-Institut der Max-Planck-Gesellschaft)

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