Operando soft-Xray spectroelectrochemistry at the MAX IV Laboratory

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HIPPIE is a high-flux, high-resolution soft x-ray beamline at MAX IV Laboratory (Sweden) with a new dedicated experimental setup for operando studies of electrochemical interfaces. Such experiments principally utilize the dip-and-pull method to form a thin liquid meniscus on the surface of the working electrode in a three-electrode cell with a liquid electrolyte solution. Both the liquid film itself and the electrode-electrolyte interface can then be probed using X-ray photoelectron spectroscopy (XPS) or X-ray absorption spectroscopy (XAS) whilst maintaining full electrochemical control. The technique can be used to probe oxidation state changes, chemical shifts, electronic structure and electrochemical potentials in-situ.

In this talk we will discuss status of spectroelectrochemical XPS/XAS using soft X-rays, including specific examples of dip-and-pull at HIPPIE has been applied to photo-electrocatalysis, battery interfaces, molecular redox reactions and metal corrosion. We will outline the experimental realities and challenges that any potential new user of the dip-and-pull method should be aware of, and additionally compare dip-and-pull to other cell designs that are compatible with soft-Xray spectroscopy.

The HIPPIE beamline operates in the 250-2000 eV range, providing access to the L absorption edges of many transition metals and the K edges of light elements. The dip-and-pull XPS experiments are realized with an ambient-pressure hemispherical electron analyzer allowing measurements in vapor pressures up to 25 mbar. XAS can be measured in several modes, including total and partial electron yield or total florescence yield. The system is compatible with aqueous electrolyte solutions as well as some organic solvents, including many of those common in batteries. An argon/nitrogen atmosphere glove box can be attached to the measurement chamber such that air sensitive materials can be studied. Typically foils or thin films are used for the working electrode. This apparatus therefore provides one of the most flexible platforms for electrochemical studies using soft-X-ray spectroscopy.

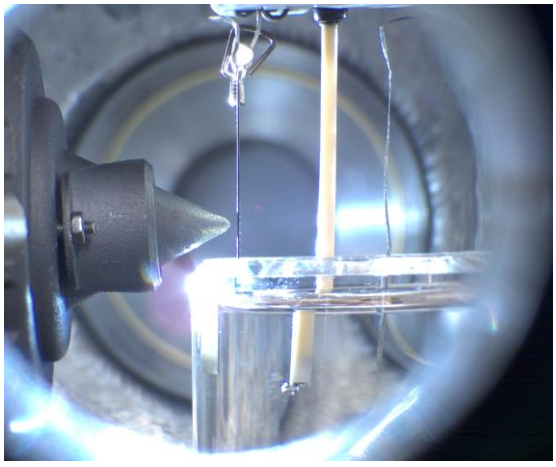

Figure 1: Photograph of inside the experimental chamber at the HIPPIE beamline during a photoelectrochemical dip-and-pull experiment. Shown is the entrance to the photoelectron analyser (left) with a three-electrode setup (suspended from above) and a beaker of aqeous electrolyte solution.