

Near-Ambient Pressure X-Ray Photoelectron Spectroscopy as a Tool for Studying Electrochemical Reactions: Opportunities and Challenges

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Résumé

Synchrotron-based Near-Ambient Pressure X-ray Photoelectron Spectroscopy (NAP-XPS) has recently emerged as a powerful tool for the investigation of electrocatalytic materials relevant to fuel and electrolysis cells during their operation. It allows one probing the interfacial polarization, detect reaction intermediates, follow red-ox transformations of species located either at the electrode or electrolyte site of the electrified interface, observe segregation/dissolution and other important phenomena.

In this presentation we will discuss various challenges specific to synchrotron-based NAP-XPS. We will touch upon « pressure gap » effects, in particular influence of the relative humidity on the electrochemical response, and ambiguities, which may be related to the attribution of the spectroscopic features. We will also compare various spectroelectrochemical cells utilized for NAP-XPS and soft X-ray absorption spectroscopy studies of electrified interfaces at ambient conditions and briefly discuss their advantages and disadvantages.

In the second part of the talk we will consider *operando* NAP-XPS and soft X-ray absorption spectroscopy data acquired during the electrocatalytic oxygen evolution reaction on Ir- and Ru-based anodes. We will consider a controversial issue of the cation *vs.* anion redox oxygen evolution reaction mechanism, discuss the nature of the active intermediates and their dependence on the type of the anode, and argue on the tentative reaction mechanism.

At the end an outlook of the future developments of electrochemical NAP-XPS will be briefly presented.

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