Scanning PhotoElectron Spectro-Microscopy – opportunities and possibilities of operando microimaging and chemical analysis.

Due to the short escape depth of electrons X-ray Photoelectron Spectroscopy (XPS) is the best surface sensitive analytical techniques for probing surface and interface chemical composition. The Scanning PhotoEmission Microscope (SPEM) uses a direct approach to add the spatial resolution and characterize materials at the submicron scale i.e. the x-ray photon beam is downsized to a submicron spot and the sample surface is mapped by scanning the sample with respect to the focused beam. With the SPEM hosted at the Escamicroscopy beamline (Elettra-Sincrotrone Trieste) the beam can be downsized, by using Zone Plates, to a diameter of up to 150 nm with overall energy resolution better than 200 meV [1].

Investigation of complex systems in electrochemistry and catalysis often requires Near Ambient Pressure (NAP) conditions. Due to samples inhomogeneity in submicron scale conventional XPS systems did not allow to examine them in proper way. We present an alternative way for operando XPS measurements based on a special NAP cell [2] (SI fig. 1) with working pressure up to 2×10^{-1} mbar and temperature range from room temperature (RT) up to 550° C. The NAP cell combined with focused synchrotron beam allows for continuous operando chemical characterization of the systems and imaging the surface within scanning areas of $450 \,\mu\text{m}^2$ [1]. It opens new opportunities for operando measurements on the systems in micro- and nanometric scale.

Fuel cells are electrochemical devices providing efficient and environmentally-friendly production of electricity directly converting the electrons exchanged in a redox reaction (such as a combustion) into electric current. One of the still unresolved issues that impedes their widespread applications is related to the limited durability of crucial components and mass transport events that deteriorate the performance.

Recent achievements in the chemical and electronic characterization of fuel cell components will be presented providing an overview of the capabilities of The NAP cell technique. For example the in situ characterization of novel non-noble metal catalysts for the Oxygen Reduction Reaction (ORR) and the characterization of a Self-Driven Single Chamber SOFC in operando condition will be shown [3].

- [1] https://www.elettra.eu/elettra-beamlines/escamicroscopy.html
- [2] H. Sezen et al. ChemCatChem, Vol. 7 22, pp. 3665-3673 (2015)
- [3] B. Bozzini et al. Scientific Report 3, 2848, 2013



SI fig. 1: SPEM layout (a), NAP cell layout (b) and cross section (c), 450x450 µm2 photoemission map (d).