In situ Scanning photoemission imaging and spectromicroscopy at near ambient pressure conditions

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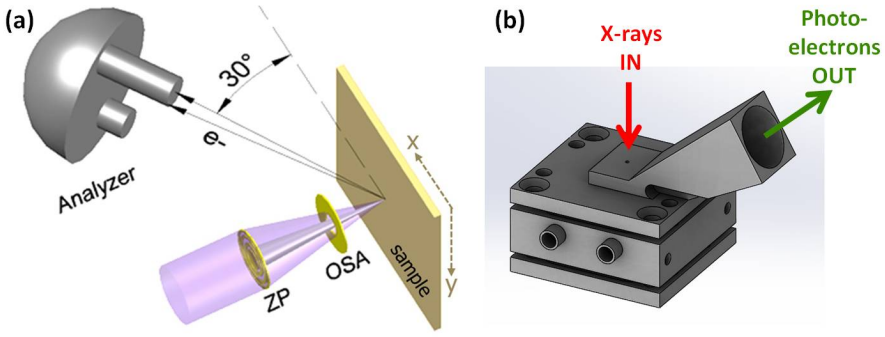
X-ray photoelectron spectroscopy (XPS) is one of the most powerful tools to characterize surfaces of materials and therefore needed for the understanding of the processes during heterogeneous catalysis. However, the standard approach of this technique suffers from two major limitations: spatial resolution and the requirement for UHV conditions.

The Scanning Photoemission Microscope (SPEM), hosted at the ESCAmicroscopy beamline at the Elettra synchrotron light source, uses a focused X-ray probe to illuminate the sample. Thus, a sample characterization at the submicron scale is possible. A Zone plate (ZP), which is a Fresnel type lens, in combination with an order sorting aperture (OSA) is used to focus the X-ray beam. The SPEM can operate in two modes: imaging and spectroscopy. In the first mode the sample is scanned while illuminated with the focused X-ray beam, and the synchronized collection of the photoelectrons with a selected energy provides a mapping of the sample. The second mode is XPS from a microspot. The X-ray beam can be downsized to a diameter of 120 nm. The overall energy resolution is better than 200 meV.[1]

To overcome the limitations of the UHV conditions a near ambient pressure (NAP) cell containing the sample can be placed within the chamber of the SPEM. The cell contains small pinholes with diameters of 200 - 400 μm fitting the geometry of the SPEM for the focused X-ray beam and the emitted photoelectrons, respectively. The impedance of these pinholes allows pressures up to 0.1 – 1 mbar within the NAP cell while maintaining a suitable pressure in the SPEM chamber. The sample can be annealed up to 600 °C using a ceramic encapsulated heater inside the NAP cell. The cell is connected to a gasline that provides various gases.[2, 3]

This unique combination of spatial resolution with near ambient pressure and elevated temperatures provides a perfectly suitable setup to investigate surfaces under reactive conditions like the oxidation of copper.[3] Recent examples of *in situ*, spatially resolved measurements under NAP conditions will be presented.



(a) Setup of the SPEM and (b) drawing of the NAP cell.

References

[1] https://www.elettra.eu/elettra-beamlines/escamicroscopy.html

[2] H. Sezen *et al.* ChemCatChem, 7, **(2015)**, 3665

[3] H. Sezen *et al.* Surf. Interface Anal. **(2017)** doi:10.1002/sia.6276