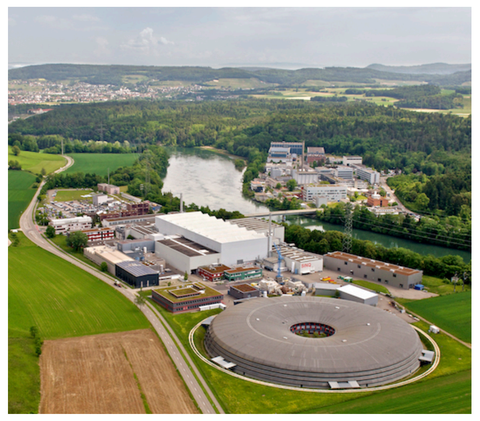
Ambient Pressure X-ray Photoelectron Spectroscopy at the Swiss Light Source

Zbynek Novotny, Nicolo' Comini, J. Trey Diulus, Jürg Osterwalder

University of Zurich[UZH]

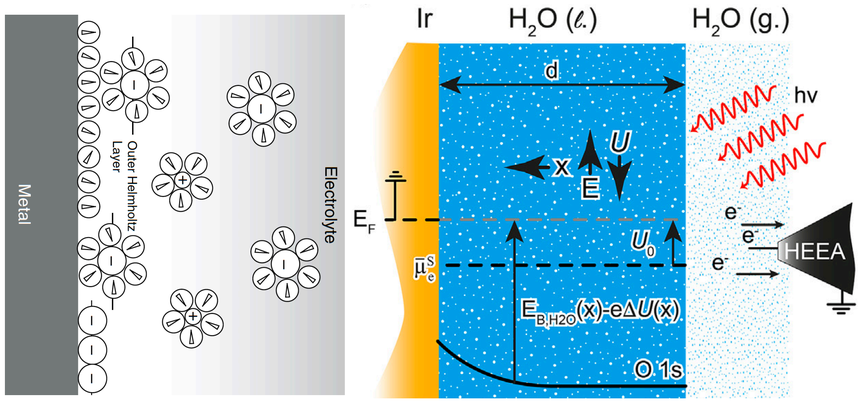


Motivation

- Osterwalder group recently set up a new ambient pressure XPS endstation at the Swiss Light Source at PSI
- Combine ambient-pressure X-ray photoelectron spectroscopy and *in-situ* electrochemistry into one instrument
- Stabilize thin electrolyte film by the dip&pull method
- Using tender X-rays, probe the properties and chemistry at the solid-liquid and liquid-gas interface
- Possibility to study solid-gas interaction on well-defined systems (e.g. Ir(001), Ru(0001))

Scientific objective: spectroscopic access to the electrochemical double layer

- The description of the electrochemical double layer is one of the foundations of electrochemistry



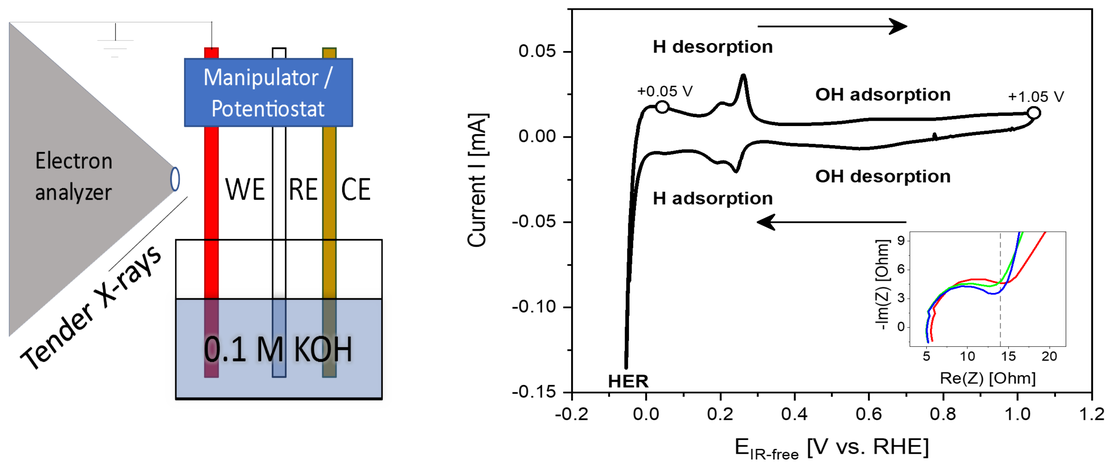
The dip&pull method can provide access to:

- the potential distribution within the double layer
- distribution of ions within the double layer
- spatial distribution of contaminants

Lichterman, M. F.; Richter, M. H.; Brunschwig, B. S.; Lewis, N. S.; Lewerenz, H.-J., *J. Electron. Spectrosc. Relat. Phenom. 221, 99 (*2017)

Dip&pull method

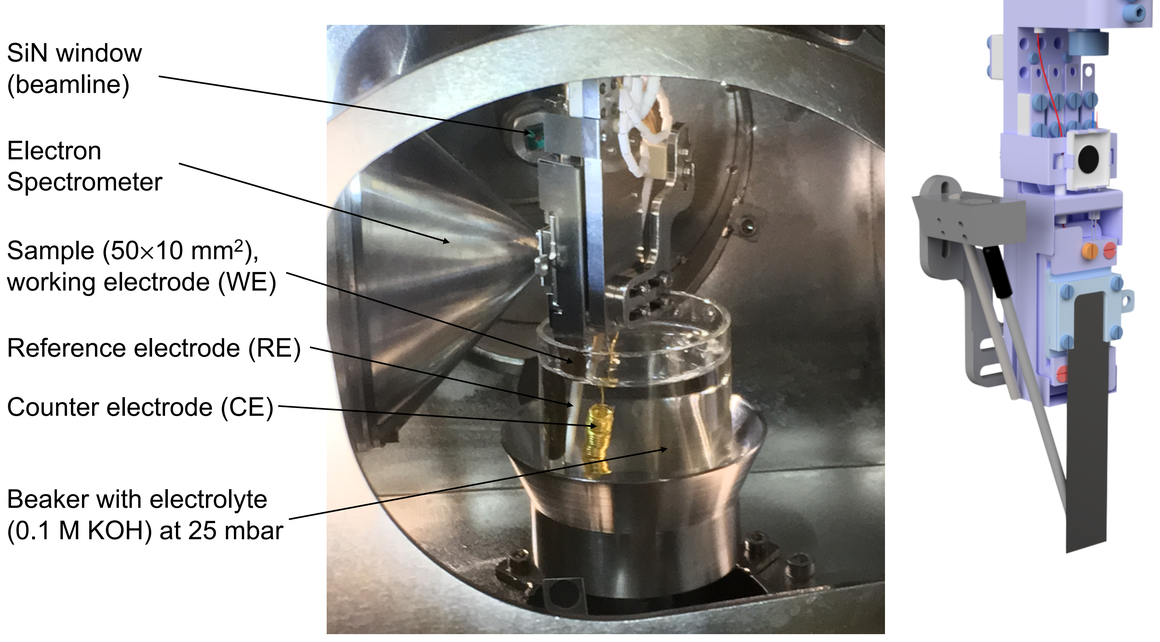
- Solid-liquid interface created by the dip&pull method:



- Prior to immersion: sample exposed to vapor at equilibrium pressure or lower
- During immersion: measure CV, sample is cleaned
- After immersion: sample pulled with a thin liquid film on the surface, with potential control

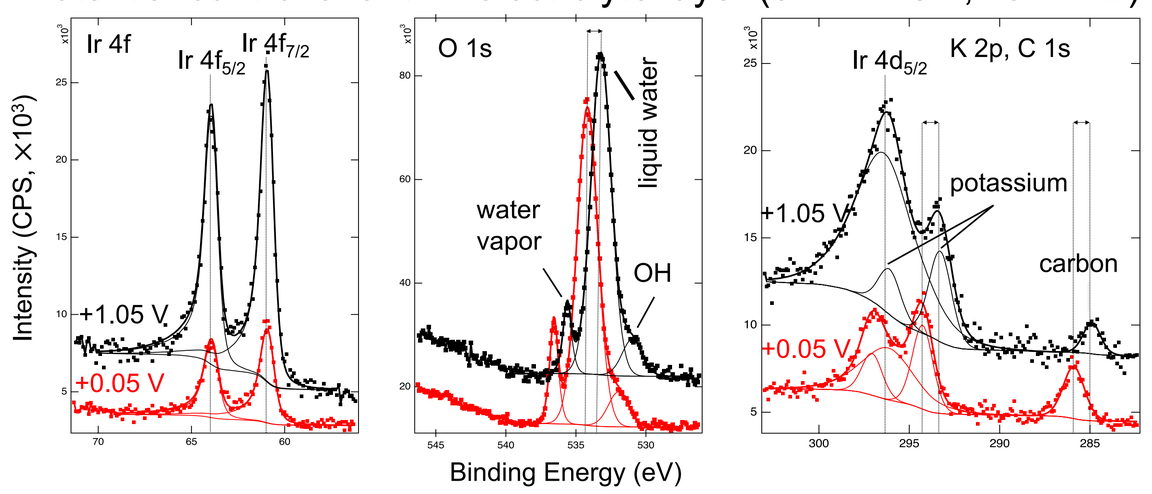
S. Axnanda et al., Sci. Rep. 5, 09788, 2015

Liquids inside vacuum chamber



Potential control within thin electrolyte layer

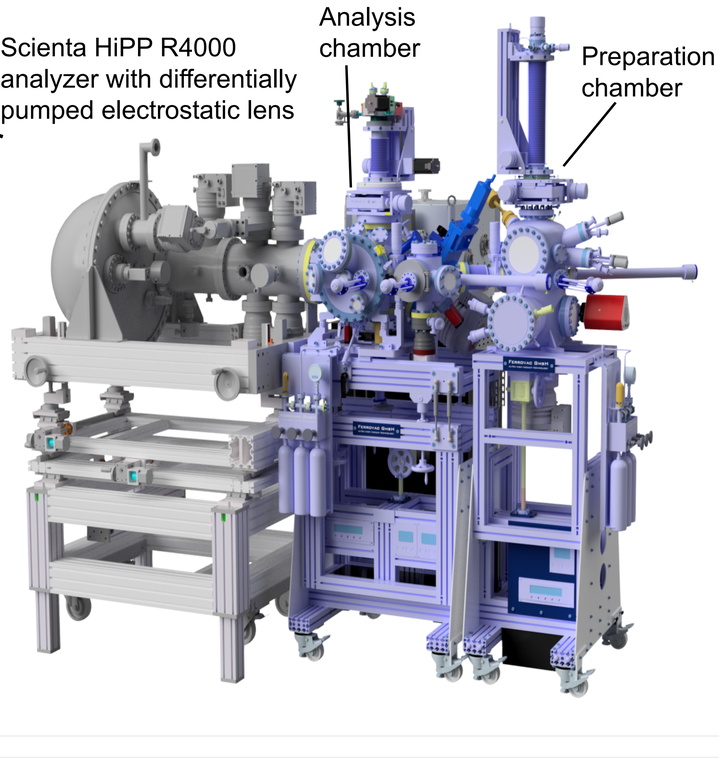
- Ir(001) WE grounded; Ir peaks do not shift with applied bias
- Potential control over thin electrolyte layer (0.1 M KOH, vs. RHE)



- With tender X-rays (here 4 keV) and electrolyte layer thickness between 20-30 nm, we can probe the solid-liquid interface for the first time using single crystalline surface

Solid-liquid interface chamber (SLIC)

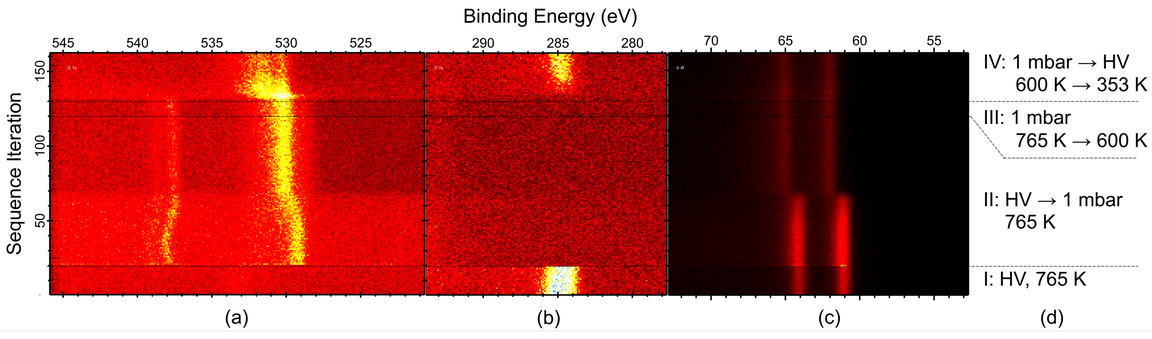
- Versatile instrument, operating in a pressure range from 1×10^{-10} mbar up to 50 mbar
- Analysis chamber: manipulator insert for solid-liquid interface experiments
- Sample preparation and characterization: evaporator, sputtering, annealing, LEED, AES



J. Osterwalder, J. van Bokhoven, M. Ammann
Funded by SNF R'Equip Grant+UZH+PSI+ETH

Oxidation of Ir(001) towards IrO_2(110)

- Studying oxidation of metals (Ir, Ru etc.) *in-situ*
- Such experiments are not possible with standard instruments



Beamlines

- Home-based at X07DB beamline (bending magnet, 270-1800 eV)
- Solid-liquid interface experiments at PHOENIX (undulator type)

Open projects

- Master's thesis: Implementation of ambient pressure capabilities into Simulation of Electron Spectra for Surface Analysis (SESSA) software package (collaboration with Prof. Wolfgang Werner, TU Wien): **requires knowledge of C programming language**)
- Bachelor's thesis: oxidation of Ru(0001) towards RuO_2(110): processing and quantification of APXPS data recently acquired
- Single atom catalysis: properties and stability of single atoms stabilized at the Fe_3O_4(001) surface under ambient pressure conditions (starting from July 2020 or later)

contacts: zbynek.novotny@psi.ch