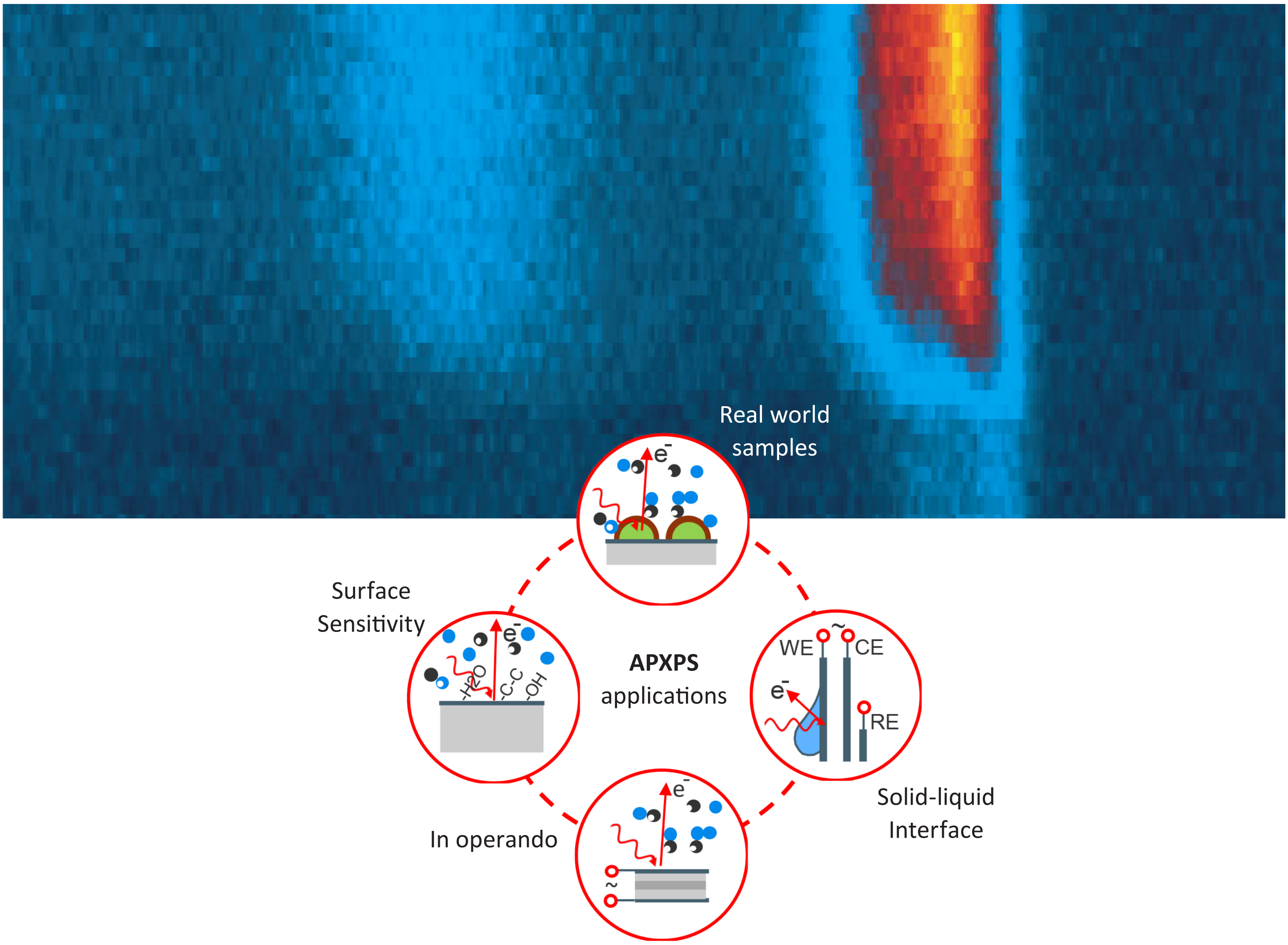
HiPP Lab

Advanced APXPS measurements made simple



- High-performance electron-analyser with transmission enhanced swift-mode
- Advanced snap-shot, imaging and angular mode
- Innovative modular design for ease of use
- Taylor made customer solutions possible

With HiPP Lab Scienta Omicron provides a novel solution for laboratory based ambient pressure X-ray photoelectron spectroscopy (APXPS). Drawing on extensive experience in the fields of photoelectron spectroscopy (PES), UHV technology, and system design, Scienta Omicron offers the HiPP Lab as an easy-to-use system that encourage user creativity through flexibility, modularity and an innovative chamber design. Scienta Omicron has a vast experience in system manufacturing in a broad range of surface science and material science applications with more than 500 PES analysers and approximately 1,000 systems deployed worldwide. This broad knowledge base has been crucial for the development of the HiPP-Lab.

The HiPP Lab is a robust, laboratory-based system that includes a powerful small-spot monochromated X-ray source and a state-of-the-art multiplex mode analyser. It is designed with ease of use and stability as the number one priority, and will serve as an efficient, turnkey workhorse in both labs and shared facilities. The HiPP Lab effectively tackles a wide range of scientific questions with applications covering catalysis, fuel cell analysis, battery research and involving the investigation of solid-gas phase, solid-liquid and gas-liquid interfaces.

HiPP-3 analyser – both stable and efficient

The heart of the system is the HiPP-3 analyser which has a proven track record with high uptime, performance and highly stable electronics. Placing two apertures at the analyser entrance, allows for reducing the number of nodes for the electrons, which is important for the electron optics. As a result, enhanced electron transmission can be achieved. Efficient pumping between the two apertures near the analyser entrance helps to remove corrosive gases which would otherwise be detrimental to the lifetime of the spectrometer resulting in reduced service cycles.

In the swift acceleration mode, a bias voltage between the first and second cones of the differentially pumped electron spectrometer is applied, which greatly enhances the transmission. At the same time, the sample is bias free. As a result, an order of magnitude gain in acquisition speed can be obtained as shown for 8.1 mbar H_2O.

Thanks to the 2D detector, angular resolved and spatial resolved measurements with < 10 µm resolution are possible. The HiPP-3 analyser is complemented by a variable spot size Al Kα monochromated X-ray source providing over 1E10 ph/s to the sample with an energy resolution of 250 meV. The spot size can be tuned to ideally match with the analyser inlet apertures of 0.3 and 0.8 mm diameter, respectively.

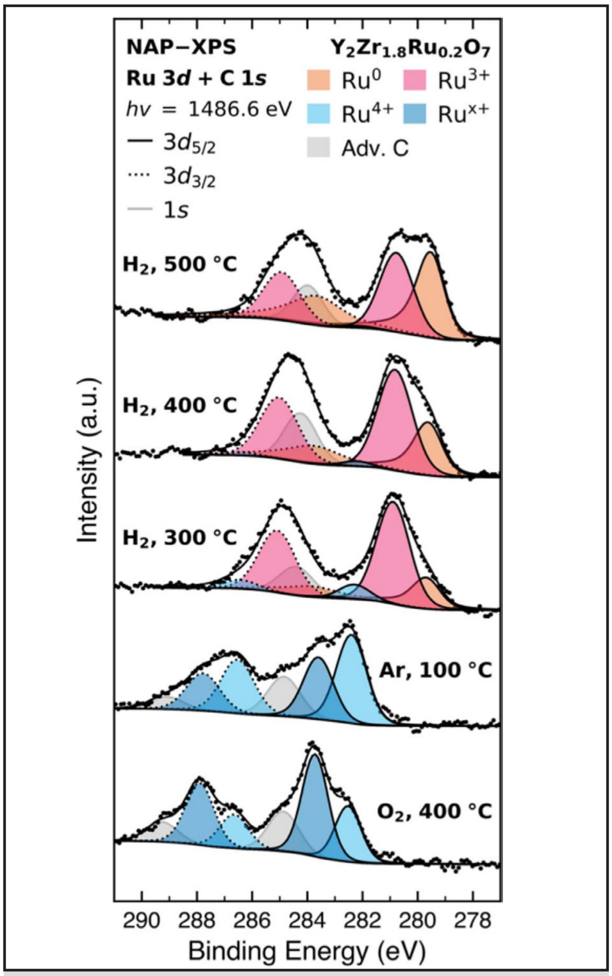


Figure 1. Photoelectron spectrum of $Y_2Zr_{1.8}Ru_{0.2}O_7$ acquired in a Scienta Omicron HiPP Lab under the following conditions and averaged over their respective activation times (t_a):
(1) 0.5 mbar O_2, 400 °C, t_a = 100 h
(2) 0.5 mbar Ar, 100 °C, t_a = 80 h (3–5) 0.5 mbar H_2, 300–500 °C, t_a = 60 h.
The evolution from oxidized to metallic Ru shows the activation of this materialby exsolving anchored nanoparticles on the oxide surface.

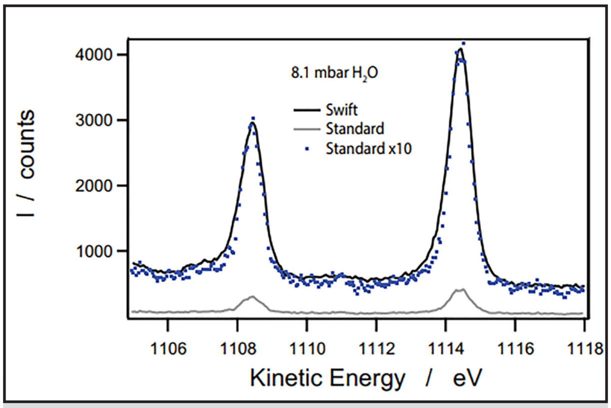


Figure 2. Al Kα excited Ag 3*d* spectra at 8.1 mbar of water recorded using standard transmission mode (grey) and swift acceleration mode (black). The transmission mode spectra has been enhanced 10 times (blue) in order to illustrate the power of the swift acceleration mode.

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Highly flexible design

The field of APXPS is rapidly evolving and new types of experimental approaches are constantly introduced. The fundamental design principle of the HiPP Lab is based on high flexibility, in order to allow for a wide range of different experimental arrangements. In Figure 3, the HiPP Lab is expanded by a glove box, a gas reaction cell, a laser heater, a preparation chamber as well as a Dip&Pull system and an electrochemical cell setup.

The functionality of the HiPP Lab can also be extended to include, e.g., automated gas flow management system with integrated mass flow controllers, Laser heating, or a UV light source.

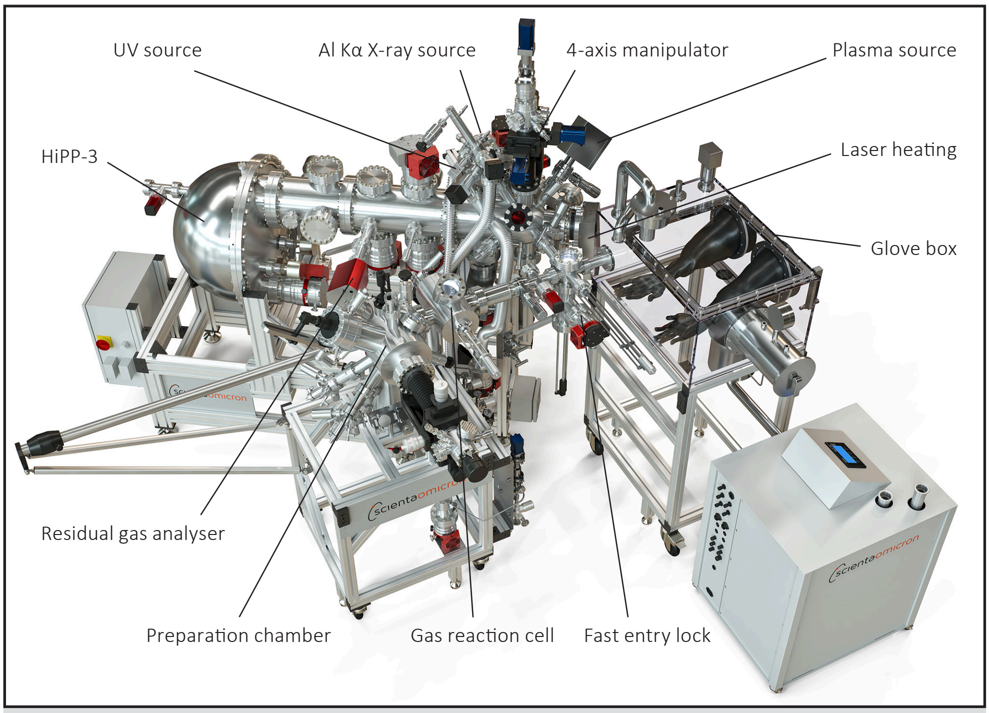


Figure 3. Example of a bespoke customer solution where the HiPP Lab is complemented with laser heating, glove box, a gas reaction cell and a preparation chamber.

The included MISTRAL software allows for system remote control of valves and turbo pumps as well as pressure and temperature reading. Together with the PEAK spectroscopy control and acquisition software the system becomes user intuitive and highly flexible to customer-wished adaptations and system expansion.

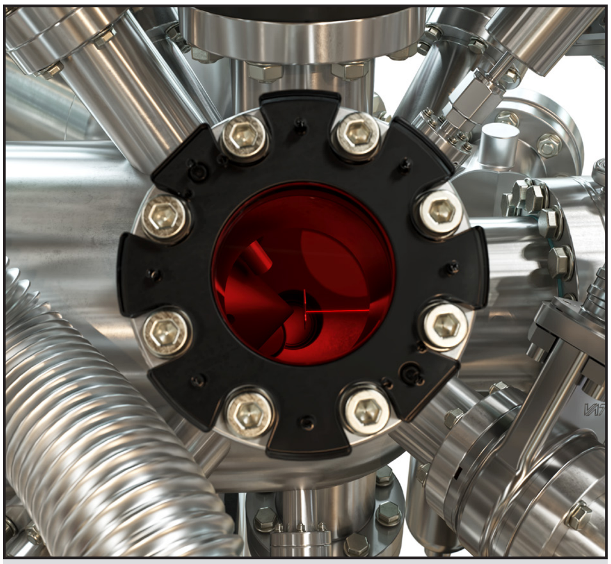


Figure 4. View into the analysis chamber of the HiPP Lab showing a representation of the flag style sample, the front cone analyser inlet, a laser protection flange and the laser heating applied to the back of the sample available as option in HiPP Lab.

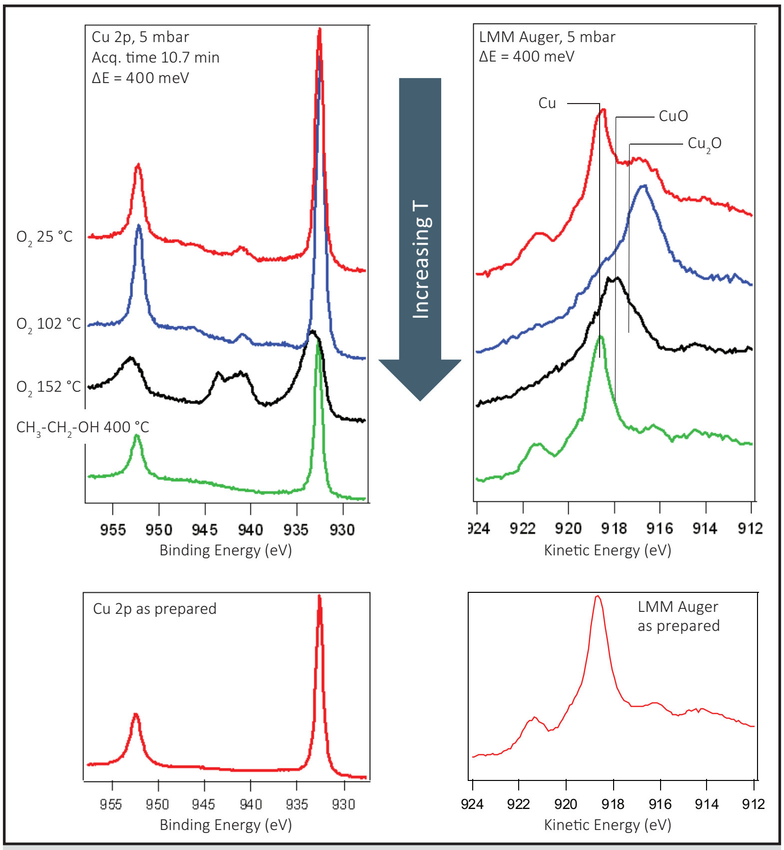
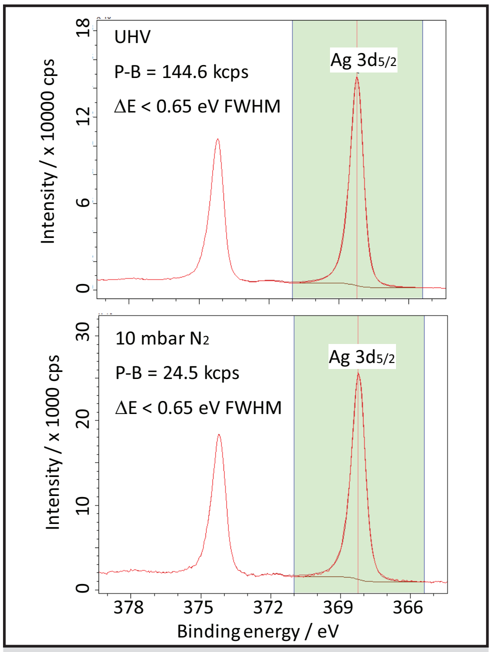


Figure 5. Oxidation and reduction study of polycrystalline copper at 5 mbar and variable temperature. The experiment starts with the as-prepared state (bottom) which is characterized by the metallic signature of the Cu. Introducing 5 mbar of O_2 gas to the system, one observes the gradual oxidation of the Cu with increasing temperature up till 152 °C. Using ca. 5 mbar ethanol and heating to 400 °C the surface is reduced back to its original metallic state.

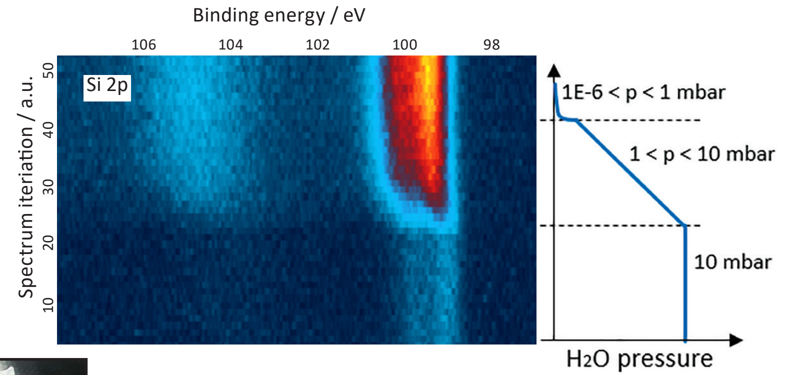


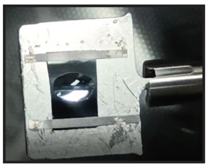
High quality Ag 3d APXPS spectra obtained using the HiPP Lab. The spectra were acquired using 0.3 mm inlet aperture and N_2 background pressure. The figure indicates the peak (P) minus background (B) intensity being 144.6 kcps at UHV, 24.5 kcps at 10 mbar, 2.2 kcps at 25 mbar and 0.4 kcps at 35 mbar

Simple and accessible with high spectral quality

The analyser and monochromatic X-ray source are mounted in the horizontal plane. This allows for a simple and accessible system with relatively moderate requirements on laboratory height and eases implementation for solid-liquid interface studies such as the Dip&Pull or liquid jet experiments. The analyser is mounted on benched rails allowing for swift exchange of analyser front cone. Thanks to the special electron-optical design of the HiPP-3 analyser, a large regime of working distances can be accommodated, making sample alignment uncomplicated.

The HiPP Lab allows for high spectral quality in all acquisition modes. The use of the snap-shot mode is eased by a homogeneous background and allows for fast spectral acquisition with temperature or pressure variations.





Front page data: APXPS spectra following the drying process of a liquid water droplet over Si in H_2O background. Spectra are acquired using snap-shot mode with Swift acceleration and a 16 eV wide energy window and 60 sec. dwell time.

Technical Data

HiPP-3 Analyser
- QPlus® AFM operation
- High electron transmission
- Long life-time and high chemical robustness
- Spatial resolution < 10 µm
- Angular mode
- Swift acceleration mode for fast data acquisition.
- Snap-shot mode combined with swift acceleration and improved homogeneous background, energy window 8 % of PE
- 2D camera- and 2D delay line detector option
- Large regime of working distance

X-ray source
- Variable spot size 250-900 µm
- Flux > 1E10 ph/s, ΔE < 250 meV
- SiN window for up to 1 bar pressure differential

Sample heating
- Resistive heating T > 650 °C in 20 mbar O_2
- Laser heating, 200 W, T > 1,000 °C in vacuum
- User selectable ramps

Software
- Mistral for user-friendly system control
- PEAK for controlling spectra acquisition

System specifications
- Guaranteed count rate for Ag $3d_{5/2}$ for FWHM < 0.8 eV, UHV 80 kcps, 10 mbar 10 kcps, 25 mbar 1 kcps, 35 mbar 100 cps

Optional Modules
- Glove box
- Gas reaction cell for sample pre-treatment at p > 10 bar and T up to 1,000 °C
- He light source
- Dip & Pull
- Electrochemical cell
- Preparation chamber with Ar sputtering, annealing, LEED, evaporator options
- Residual gas analyser
- Automated gas flow management system with integrated mass flow controllers

Learn more at **www.scientaomicron.com/HiPP-Lab**