HIGH PRESSURE (HP) XPS
AMBIENT PRESSURE XPS (ESCA) - SYSTEM

PRECISION AND VACUUM TECHNOLOGY

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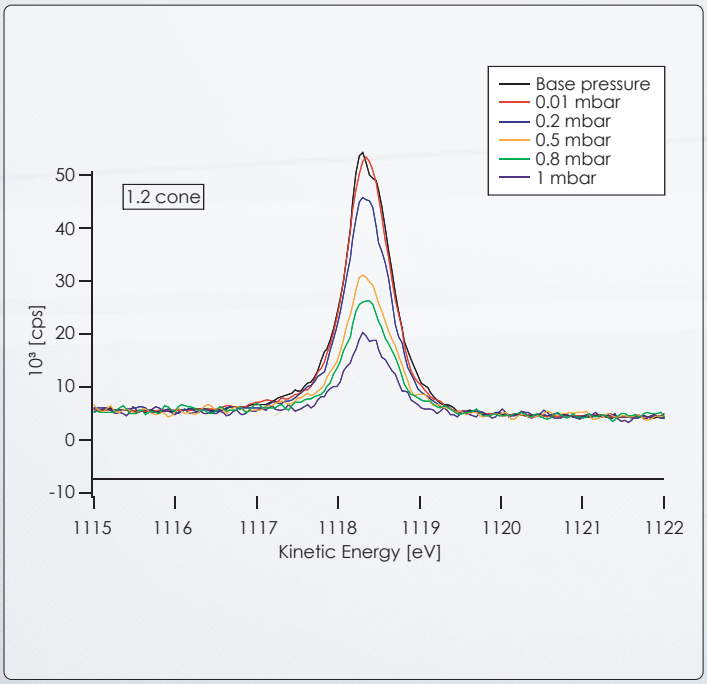
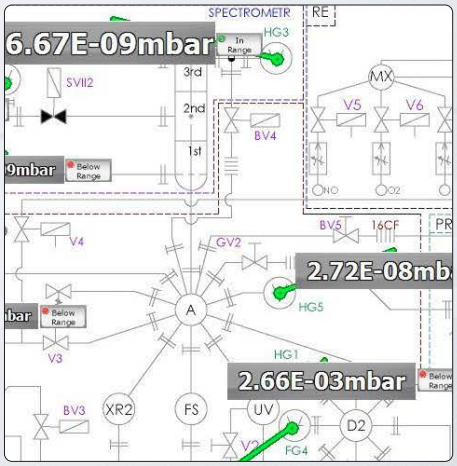
DESCRIPTION

Dedicated system for ambient pressure 1 mbar – 10^{-10}mbar XPS (ESCA) / UPS experiments with controllable sample temperature from 100 K to 850 K in analysis chamber. Full PLC protection and software control including a clear visualisation of machine state. Data acquisition and control of all integrated devices, supplies and ancillary equipment.

FEATURES

- Ambient pressure chamber 1 mbar - 10^{-10}mbar
- Equipped with HP analyser
- Differentially pumped X-ray monochromator
- Differentially pumped double anode X-ray source
- Differentially pumped UV source
- Four pumping stages for main chamber and analyser
- Integral halogen bakeout of analytical chamber
- Laser and resistive heating of sample holder
- Wide range of sample temperature: 100 K - 2200 K
- Sample preparation chamber
- High pressure reactor - up to 20 bar, 650°C
- Gas dosing system with ability of heating and thermal stabilisation
- Intro with preliminary heating
- Sample park chamber which allows storage up to 5 sample holders
- TDS sample measurement during XPS (ESCA) / UPS in ambient pressure

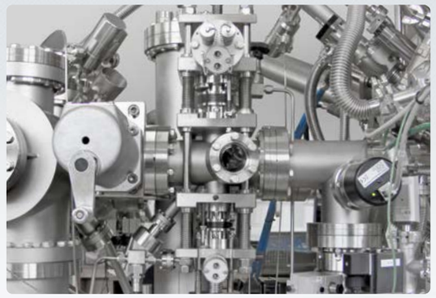




Software provides control of the system, visualisation of machine state, data acquisition, possibility to control other devices connected to the system like UV power supply, etc. System is controlled and protected by PLC controller.

X-ray monochromator

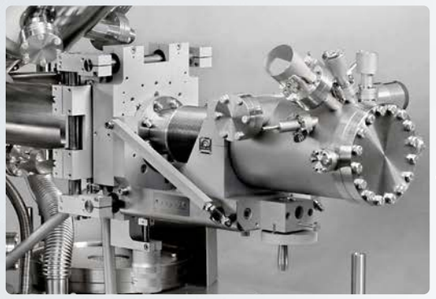
High pressure reactor

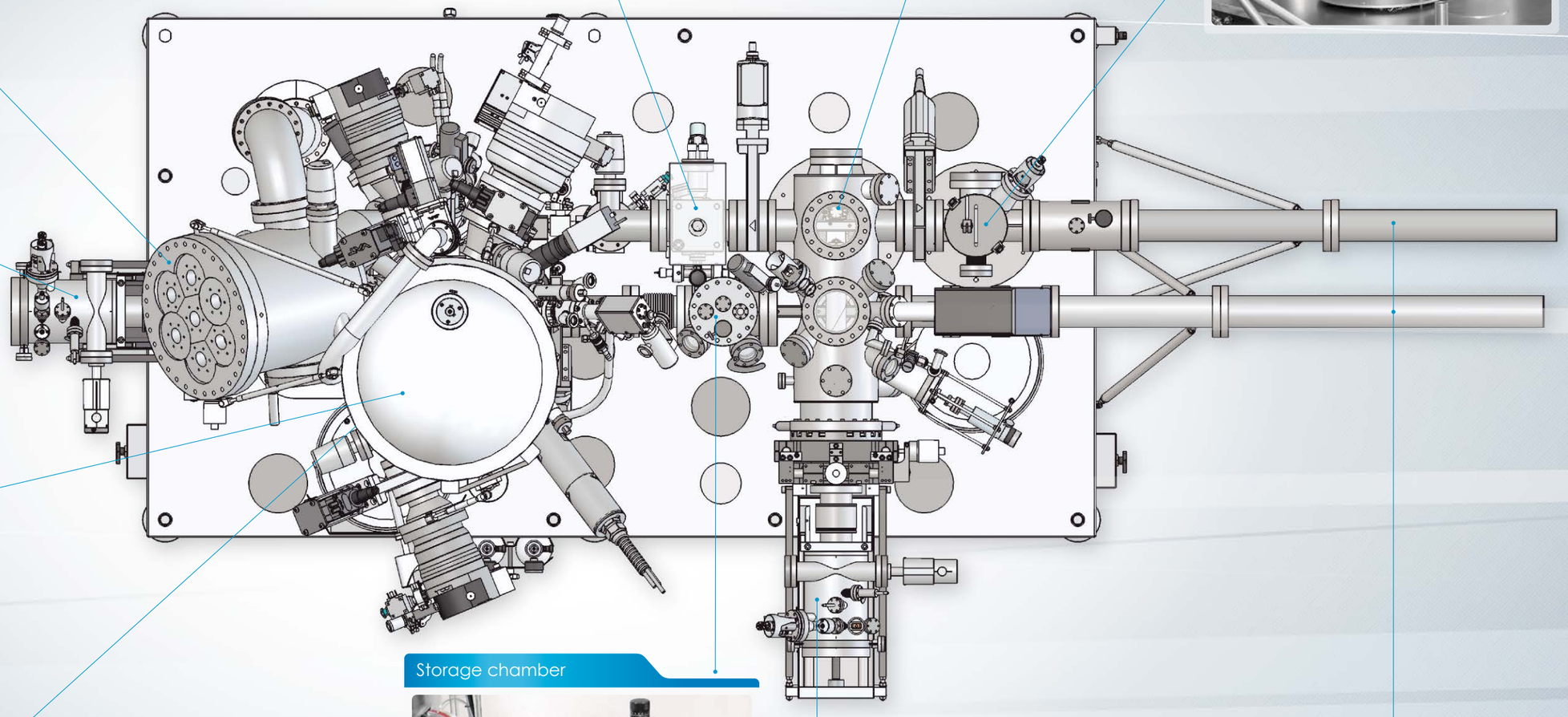


Preparation chamber

Load-lock chamber

4 - 5 axes manipulator





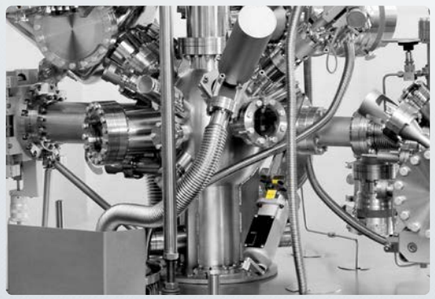
Ambient pressure analyser

Measurements at 1 mbar with analyser:

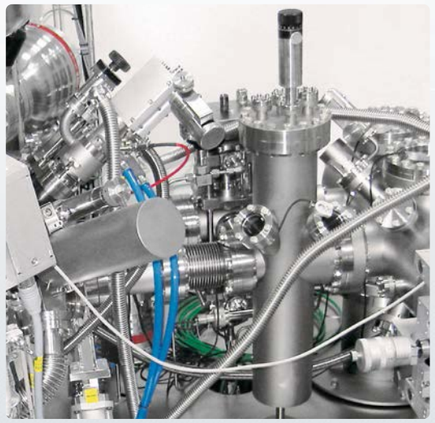
Silver, Ag3d5/2

- Energy resolution: 0.42 eV
- Working pressure: 1 mbar
- Max. Power: MX for small spot: 200W
- Roland Circle Radius: 650mm

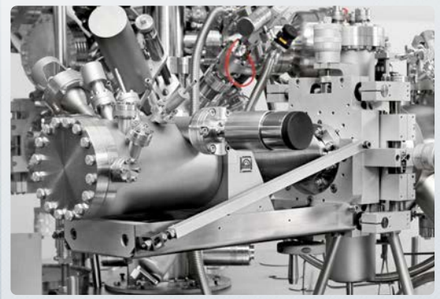
Ambient pressure analysis chamber



Storage chamber



4 - 5 axes manipulator



Linear transfers

Innovative results of cutting edge researches achieved on PREVAC's High Pressure XPS (ESCA) system.

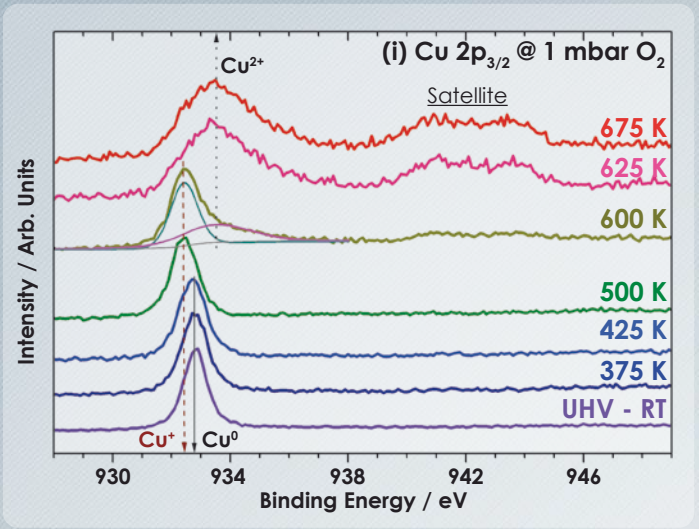
Mapping of Copper Oxidation States as a function of temperature at 1 mbar O_2

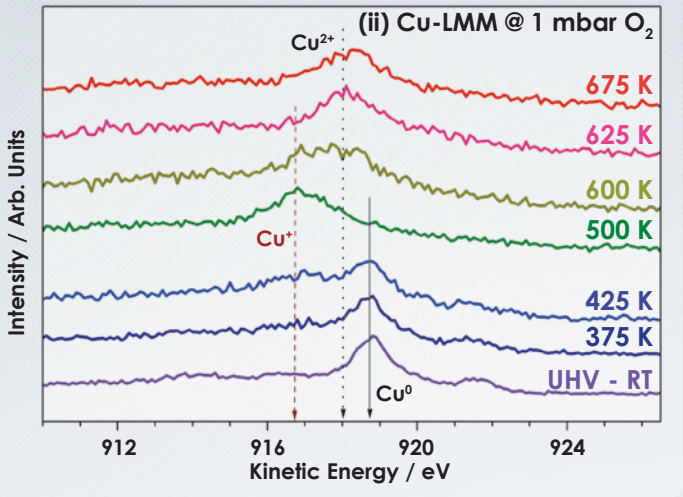
The Cu 2p, Cu LMM Auger level and valence band (VB) spectra recorded in the presence 1 mbar O_2 from RT to 675 K are shown in the attached Figures. The Cu 2p spectrum from a clean Cu surface obtained under UHV conditions and at RT immediately after Ar^+ sputtering and annealing is shown for reference (bottom-most trace in all Figures). The Cu $2p_{3/2}$ core level spectrum centered at 932.8 eV (grey solid arrow) which is characteristic of metallic Cu remains unchanged until 425 K indicating that the metallic nature of Cu surface is retained until this temperature. Cu-LMM also shows corresponding feature at 918.8 eV.

On increasing the temperature to 500 K, a dramatic change in oxidation state to Cu^+ (ie. Cu_2O) is evident from the shift observed in Cu 2p (to 932.4 eV), Cu-LMM (to 916.7 eV) compared to metallic Cu features. A distinct feature appears at 1.2 eV with a narrowing of VB feature.

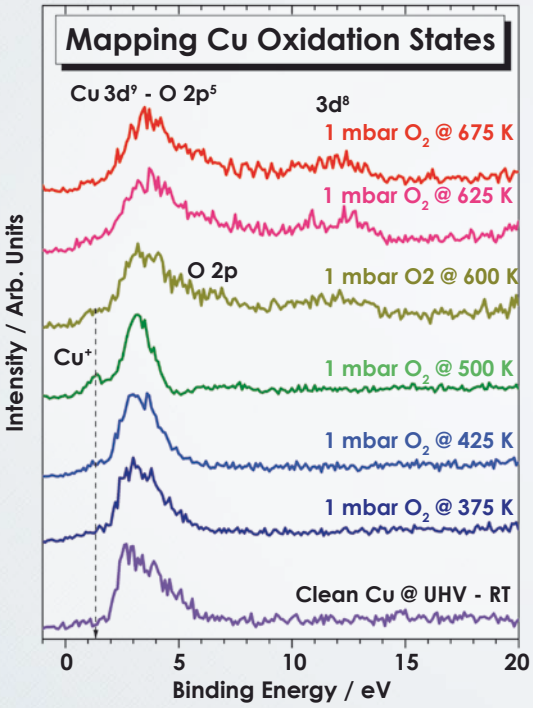
Further increase in temperature to 600 K shows features due to Cu_2O as well as CuO. This marks the beginning of oxidation of Cu_2O to CuO. A prominent satellite feature, typical for CuO ($Cu2^+$ oxidation state), begins to appear along with a shift in Cu-LMM feature. Broadening of VB with O 2p features confirms the oxidation of Cu_2O to CuO.

Spectra recorded at 625 and 675 K fully supports the complete oxidation of Cu surfaces to CuO, and sufficiently thick CuO layers observed suggest the beginning of bulk oxidation towards CuO. Features typical for CuO were observed in all three spectra, such as $3d^8$ satellites in Cu 2p core level.





Core level spectra measured while exposing a polycrystalline Cu foil to 1 mbar O_2 at various temperatures. (i) Cu 2p, and (ii) Cu LMM.



High pressure valence band spectra recorded at 1 mbar O_2 and at different temperatures. Systematic conversion of Cu metal at UHV-RT to Cu_2O (at 500 K) and CuO above 500 K is observed in 1 mbar O_2.

Ref.: Kanak Roy, C. P. Vinod, Chinnakonda S. Gopinath,* Design and Performance Aspects of a Custom Built Ambient Pressure Photoelectron Spectrometer Towards Bridging the Pressure Gap: Oxidation of Cu, Ag, and Au Surfaces at 1 mbar O_2 Pressure, *Journal of Physical Chemistry C* **117**, 4717-4726 (2013).
