Surface analysis facilities at CERN

Marcel Himmerlich on behalf of the TE-VSC-SCC team

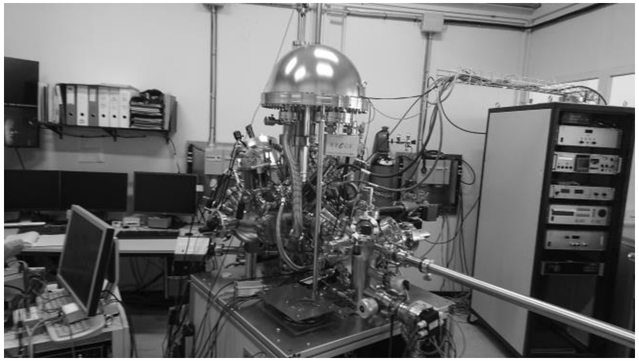
Surface Analyses – Quality Control and R&D

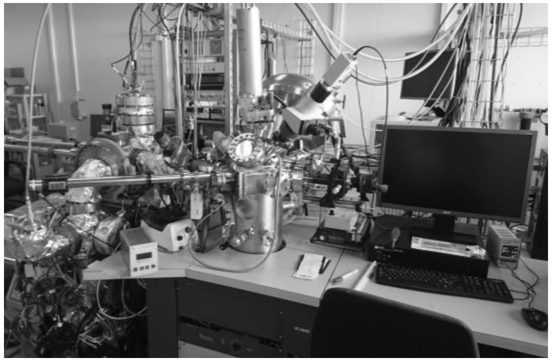
The Surface Analysis team provides service material analyses and contributes to CERN R&D and scientific studies.

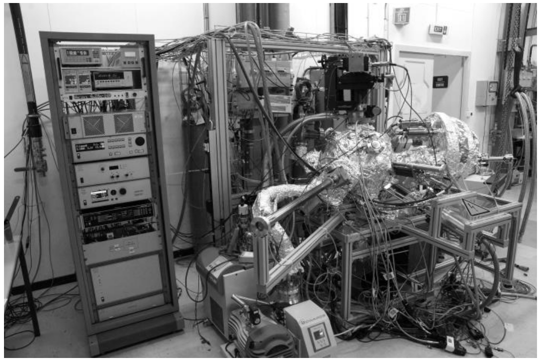
We operate UHV systems for surface spectroscopy and thin film characterization on small test samples.

Main methods:

- Secondary Electron Yield (SEY) measurements
- X-ray Photoelectron Spectroscopy (XPS)







Surface Analyses – Quality Control and R&D

- We provide surface and thin film analysis expertise for the CERN community and projects.

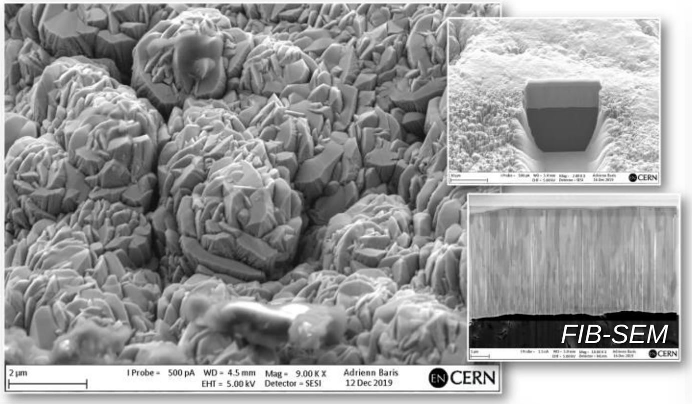
- If a surface or thin film analysis technique does not exist at CERN
  → we advise and mediate tests at external partners and institutes

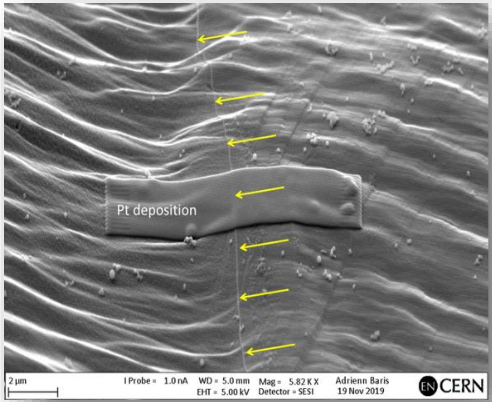
Examples: Secondary Ion Mass Spectroscopy profiling &
Ion Scattering Spectroscopy for Hydrogen content in thin metal films

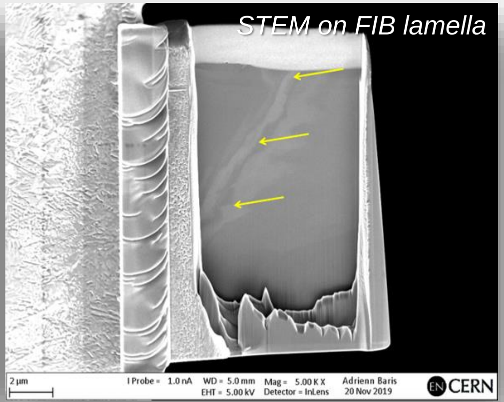
Laterally resolved Auger Electron Spectroscopy depth profiling
for surface composition analysis of superconducting cables

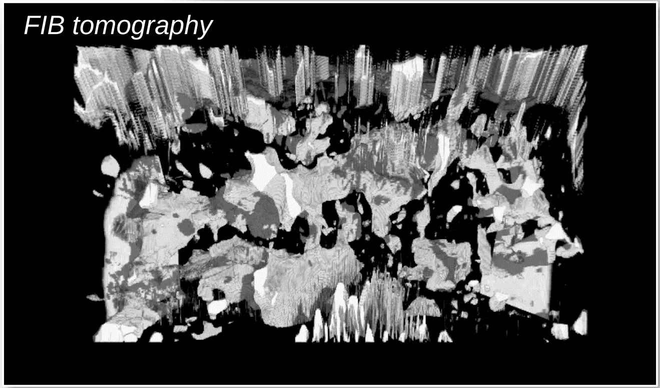
Advanced Microscopy & Diffraction @ EN-MME-MM

Topographical imaging, morphology, phase identification & chemical analysis:
FEG-SEM, FIB-SEM, STEM, EDS, EBSD,
3D FIB tomography, Automatic Particle Analysis & XRD









Analysis techniques at TE-VSC-SCC Chemistry lab

FTIR spectroscopy

*e.g.: Evaluation of surface cleanliness for **UHV applications***

Optical Emission Spectroscopy

*e.g.: **Lead** detection in CERN buildings*

Differential Scanning Calorimetry

*e.g.: Epoxy Resin **Tg Measurement***

Atomic Absorption Spectroscopy

*e.g.: Superconducting cable **composition***

Potentiometric titration

*e.g.: Quality control of **surface treatments bath***

X-ray spectroscopy

*e.g.: In-situ **Thickness** measurement of coatings*

Karl Fischer Coulometry

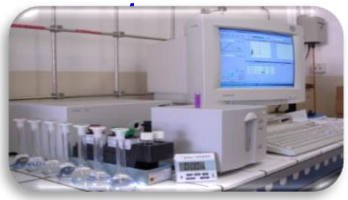
Gas Chromatography

*e.g.: Quality control of the **LHC experiments cooling fluid***

Thermogravimetry

*e.g.: Mineral filler quantification in **polymers***

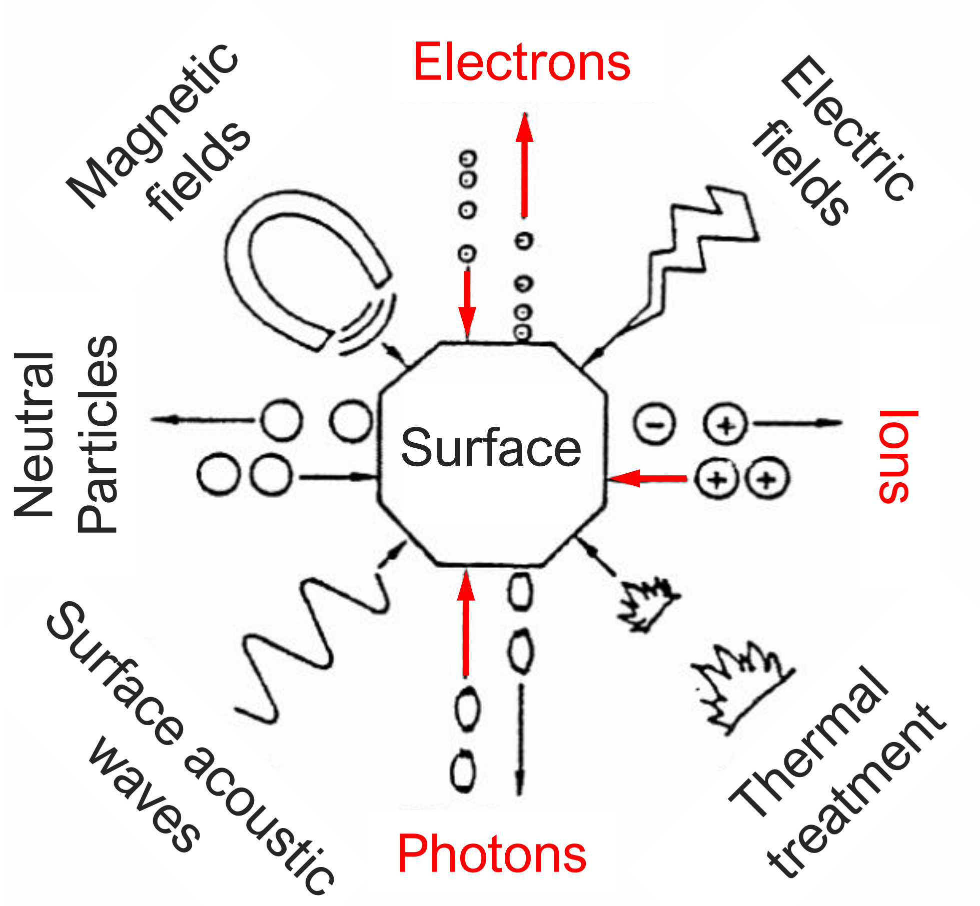
UV-Visible



*e.g.: water analysis from **STEP***

Experimental Techniques

The «playground» of surface analysis



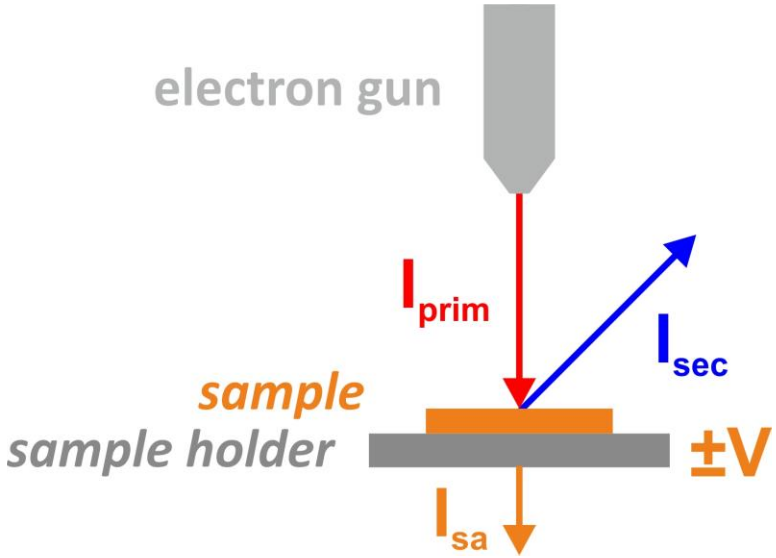
Laterally resolved analysis
→ Microscopy

Spectrally resolved analysis
→ Spectroscopy

Combination of lateral magnification and spectral selection
→ Spectromicroscopy
→ Microspectroscopy

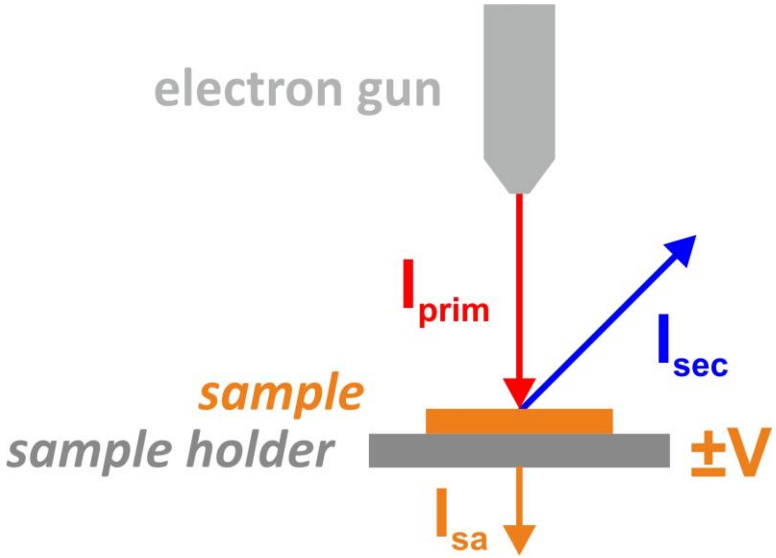
adapted from M. Henzler and W. Göpel, Oberflächenphysik des Festkörpers

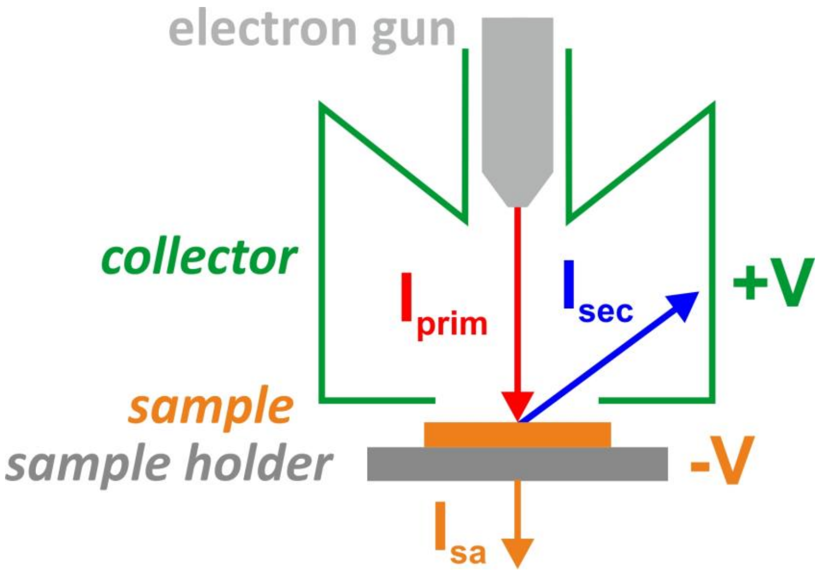
Angle-integrated **S**econdary **E**lectron **Y**ield



$$\delta(E) = \frac{I_{sec}(E)}{I_{prim}(E)} = \frac{I_{+V} - I_{-V}}{I_{+V}}$$

Angle-integrated **S**econdary **E**lectron **Y**ield

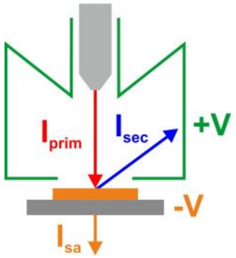


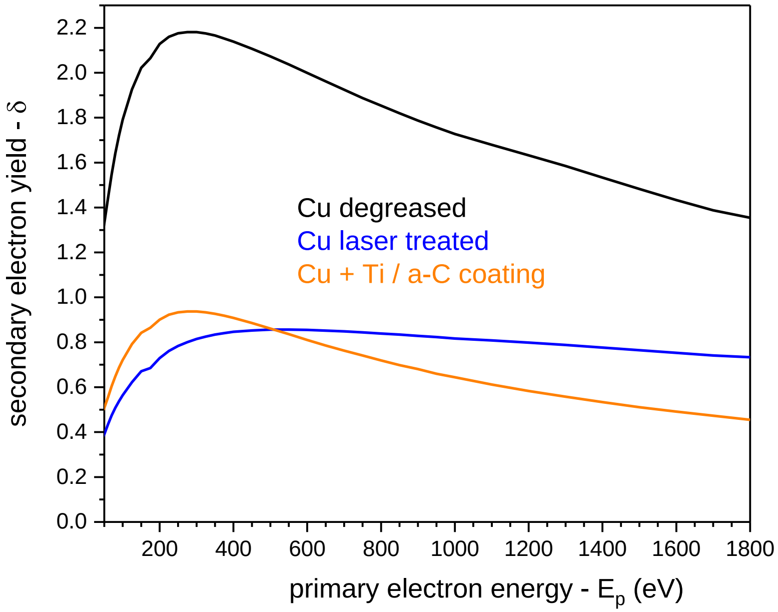


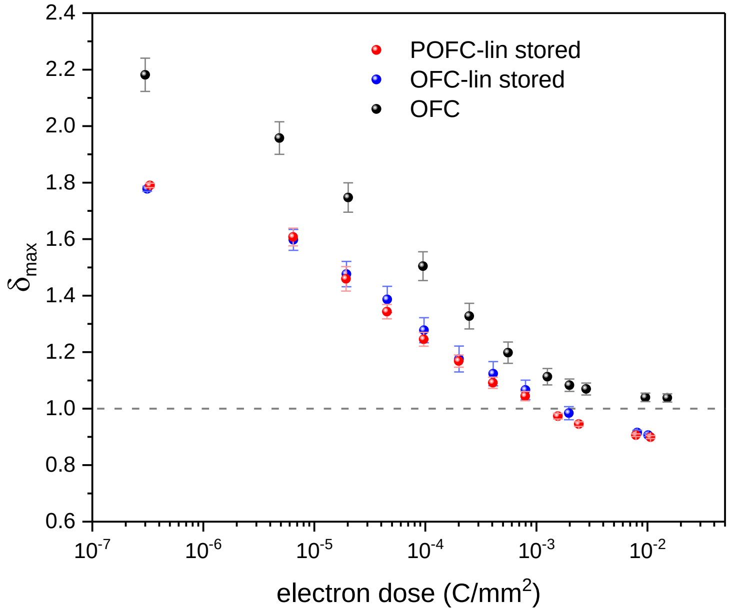
$$\delta(E) = \frac{I_{sec}(E)}{I_{prim}(E)} = \frac{I_{+V} - I_{-V}}{I_{+V}}$$

$$\delta(E) = \frac{I_{sec}(E)}{I_{prim}(E)} = \frac{I_{col}}{I_{col} + I_{sa}}$$

Secondary Electron Yield & Conditioning







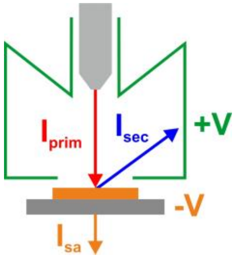
Other options:

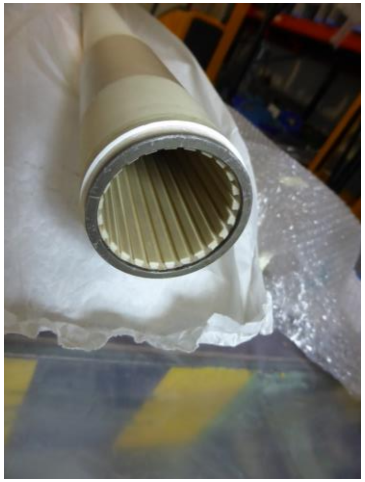
- Electron induced surface modification (conditioning)
- Work function measurements
- Incidence angle dependent SEY analyses
- SEY measurements of insulating samples

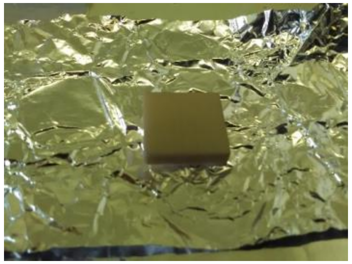
Pulsed SEY for Insulators

Problem: - Difficulties to measure continuous-current SEY on insulators due to surface charging

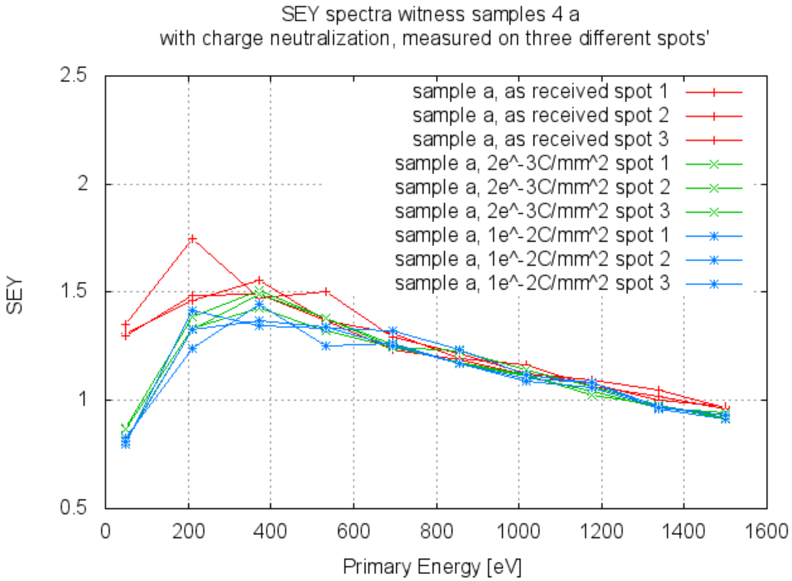
Solution: - Exposure to 30 µs long electron pulses (2×10^{-12} C per data point) on a spot of about 10 mm^2 to minimize charging
- Time-resolved measurement of primary and secondary electron current
- Neutralization with low energy electrons after each pulse to compensate positive charges





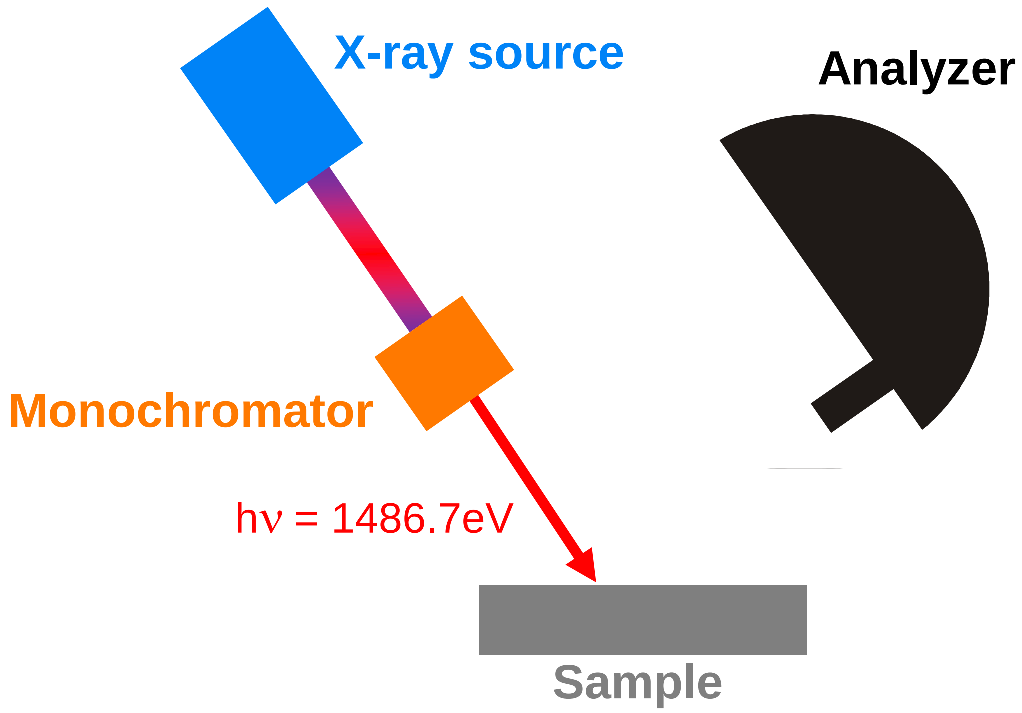


Coated Ceramic chamber for LHC injection kicker magnets and witness sample (Photos courtesy M. Barnes SEY_861)

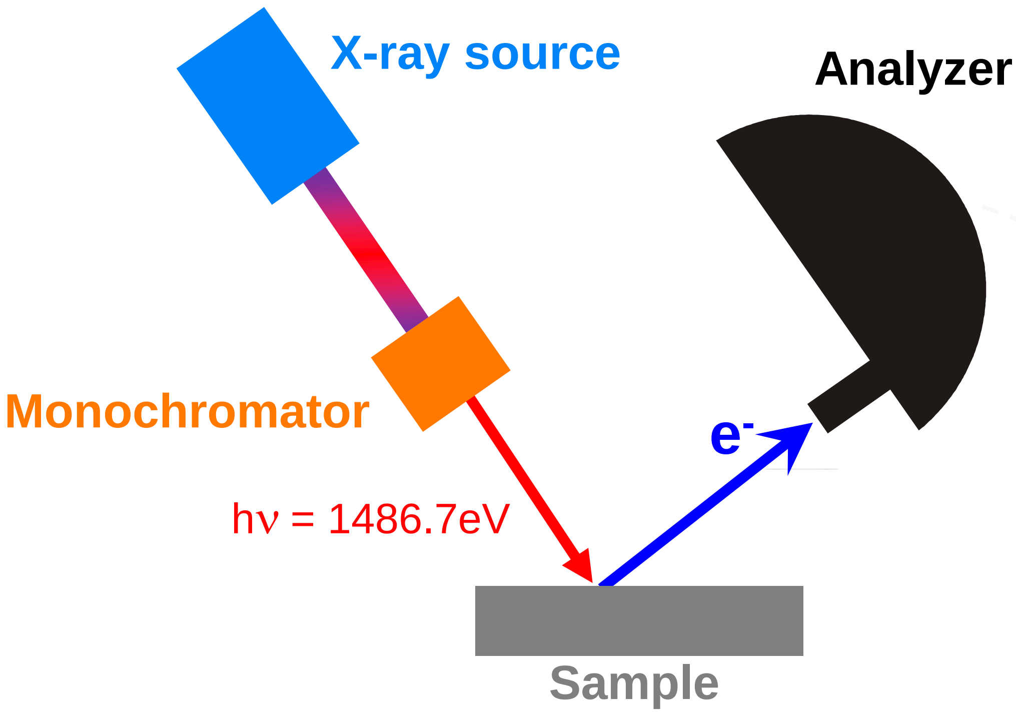


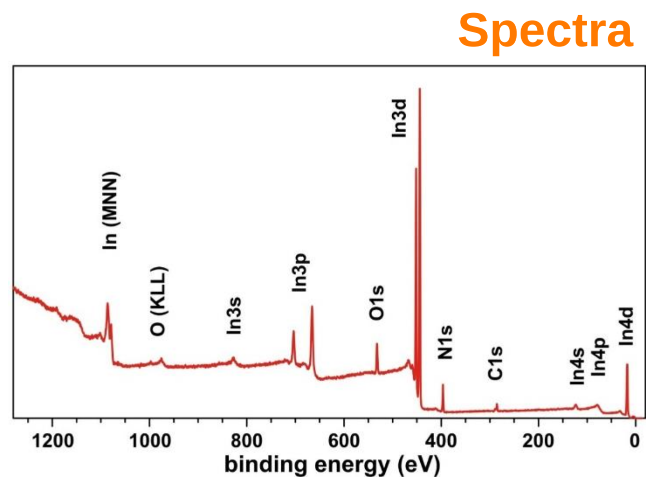
SEY spectra on witness sample with two conditioning steps

X-ray Photoelectron Spectroscopy



X-ray Photoelectron Spectroscopy





Energy distribution of emitted photoelectrons

↓

Represents the element-specific electron distribution

Results from **P**hotoelectron **S**pectroscopy

Surface chemical composition

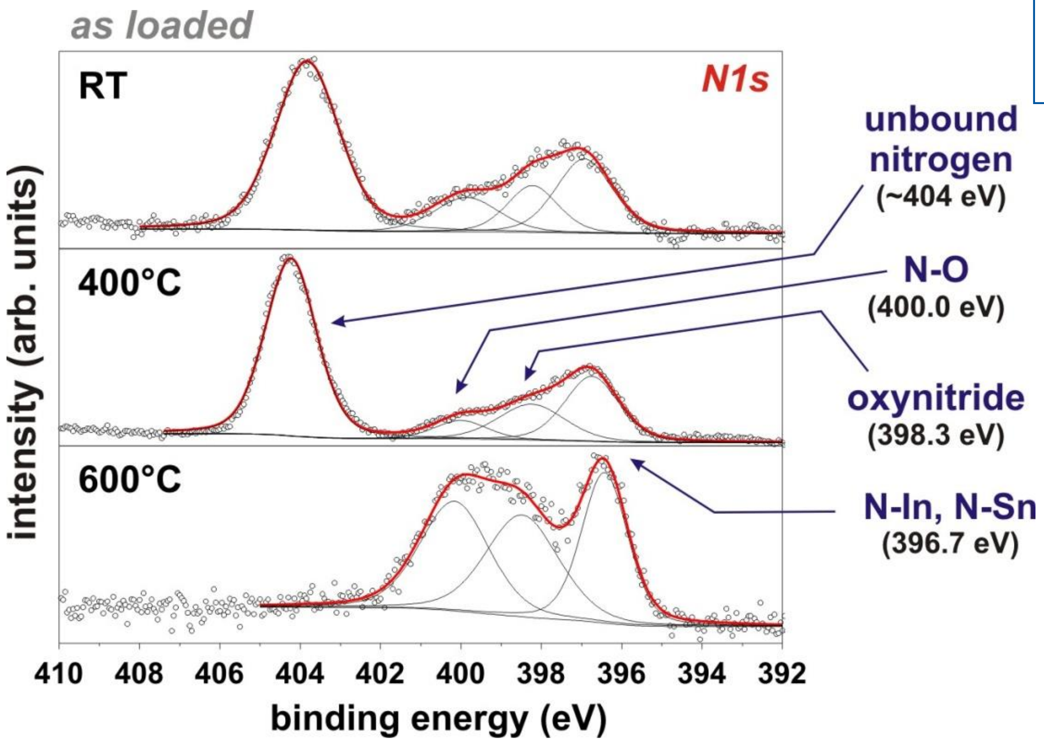
- Surface and film stoichiometry (elemental composition)
- Adsorbates, contamination & surface functionalization
- Chemical modification of surfaces
- Degradation & passivation

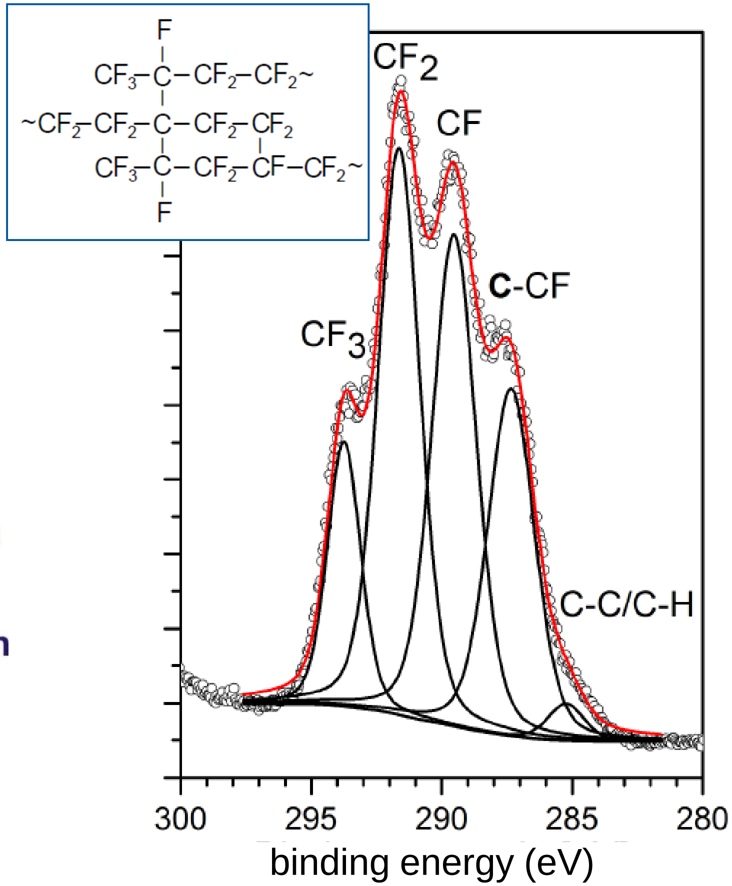
Surface electronic properties (in-situ studies)

- Valence band density of states
- Work function, surface band bending, surface dipoles
- Band offsets at interfaces
- Charge transfer processes

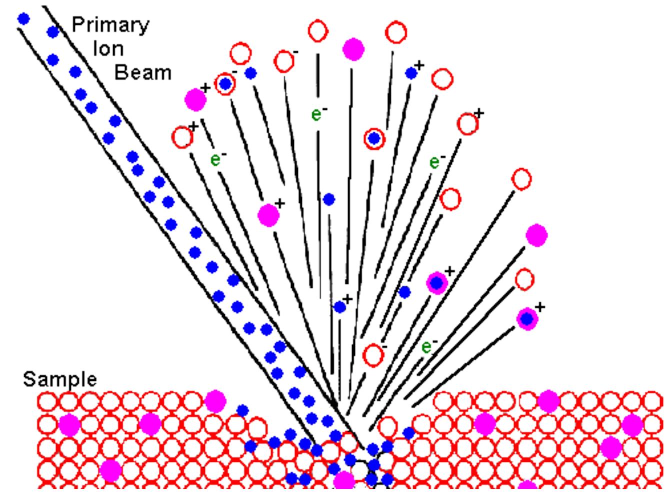
Analysis of chemical states

Electron binding energy depends on the actual chemical bond configuration of the material and the nature & electronegativity of the neighboring atoms

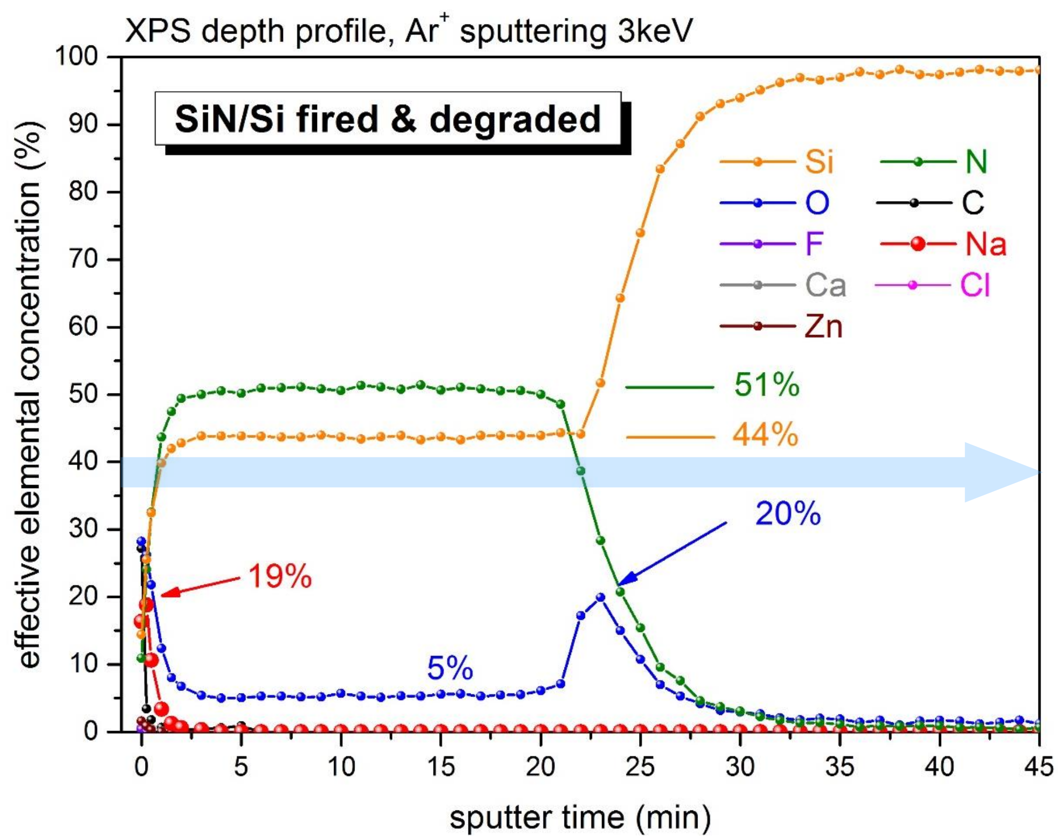




Sputter depth profiling



http://www.eag.com

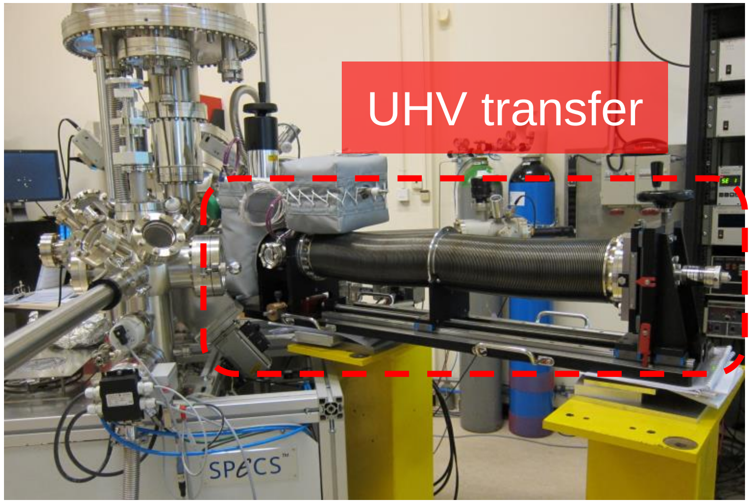


XPS: Practical and technical aspects

- Depth of information in XPS: 5-10 nm
- Detection limit 0.01 – 0.1 at.%
- Lateral resolution limited to 200 µm
- Depth profiling up to 2 µm
- Samples must be vacuum-compatible (low vapor pressure, no pencil marks, no fingerprints, contamination- and oil-free)
- Flat samples preferred & maximum sample thickness ~1 cm
- Minimum sample size 4×4 mm^2, maximum 45×45 mm^2

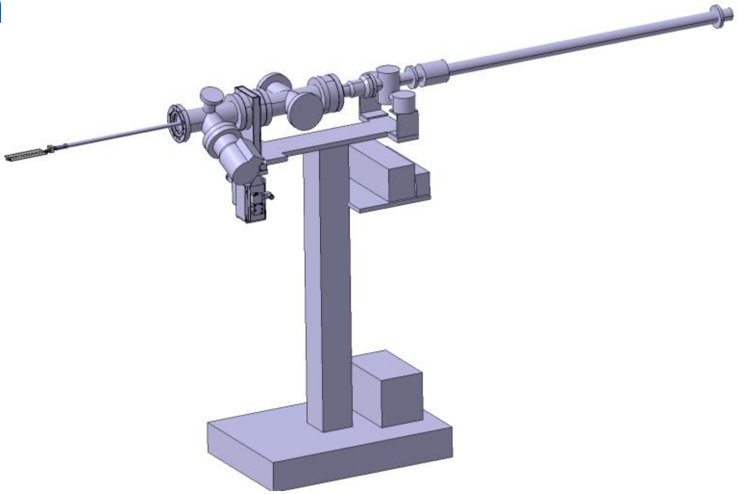
- Hydrogen not directly detectable
- Some organic materials are sensitive to X-ray damage
- Insulating samples: limitations in analysis of bond configuration

Vacuum Transfers for Surface Characterization



Characterization of Photocathodes with SY-STI-LP

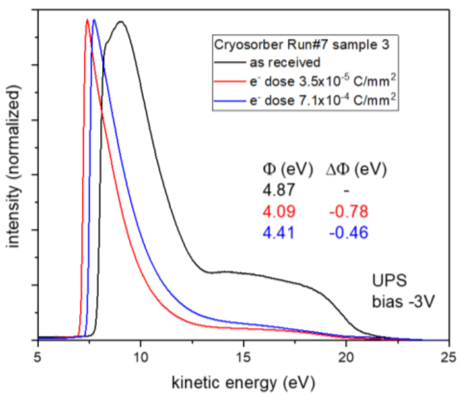
- ✓ Vacuum transfer systems for sensitive and reactive samples are essential to avoid surface oxidation and adsorption of ambient species
- ✓ Solutions exist for analysis of photocathodes, samples from the SPS to study beam-surface interaction



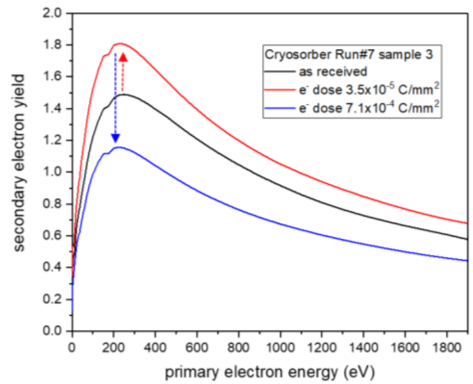
Vacuum transfer solutions can be adapted to new needs and geometries.

UV or e$^-$ induced electron spectroscopy

- UV source to measure work function and valence band



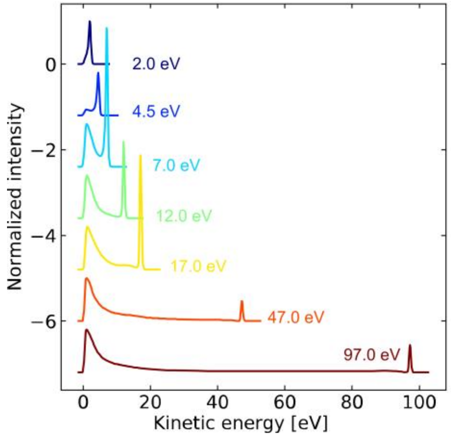
$\Phi \leftrightarrow \delta$

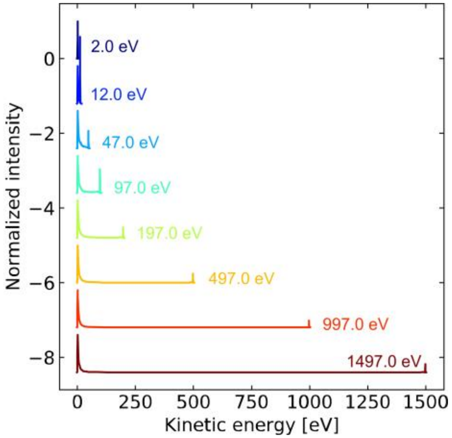


E_{photon}

21.2 eV
&
40.8 eV

- Electron source to measure angular-resolved SE spectroscopy



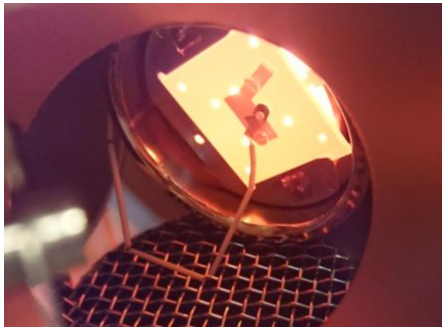


$E_{electron}$

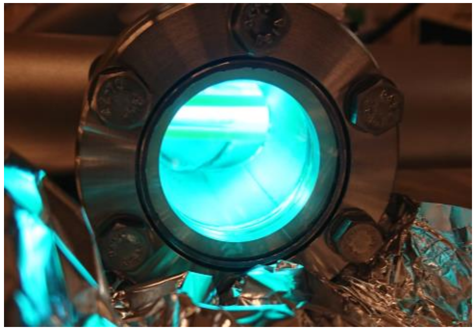
1 eV
-
1500 eV

Sample preparation & surface modification

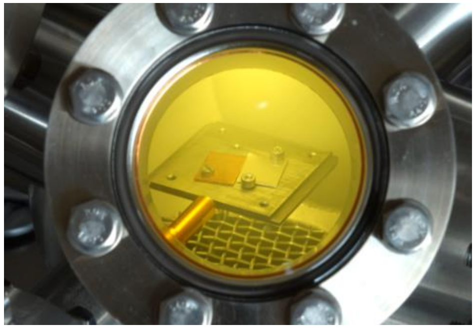
- Characterization of surface property changes during treatments in vacuum and/or reactive atmosphere (p ≈ 10^{-2} mbar)



Heating up to 800°C



UV-C irradiation



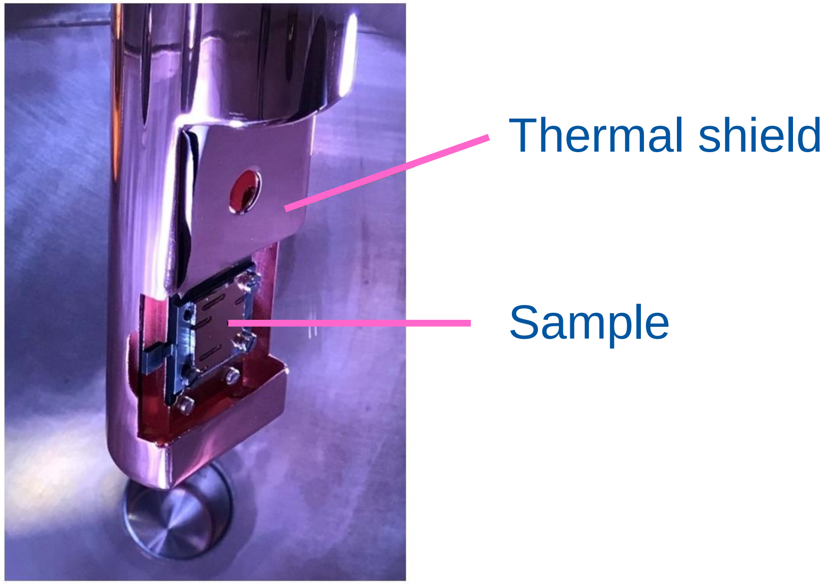
Plasma exposure

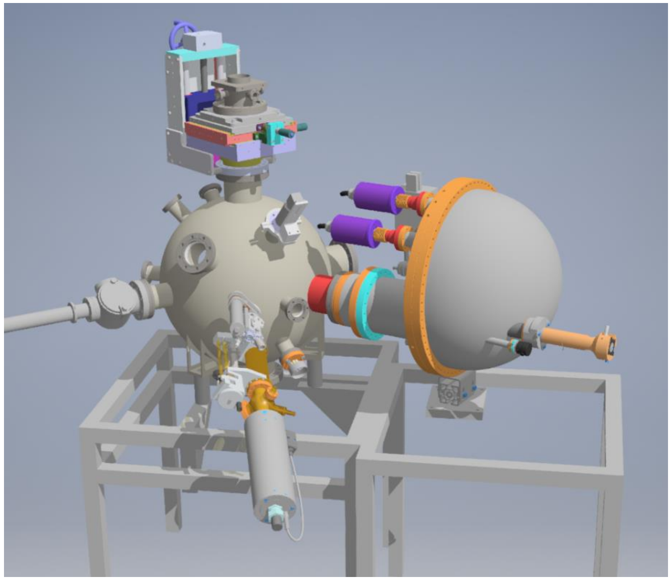
+ injection of gaseous species

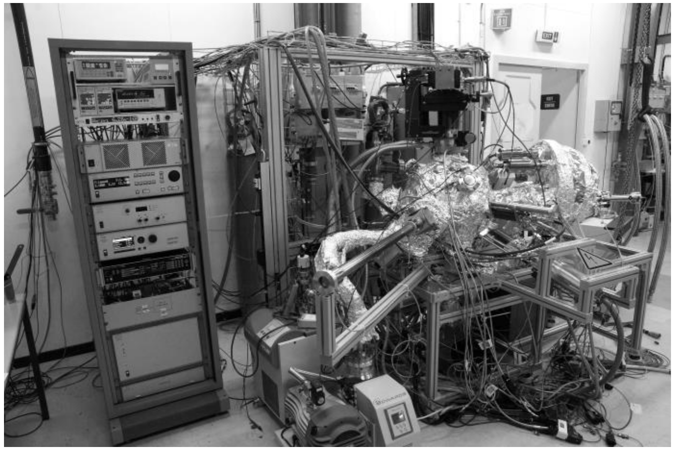
Characterization of:

- Chemical reduction/oxidation and functionalization processes
- Physical cleaning/etching of surfaces

Surface analyses at cryogenic temperatures







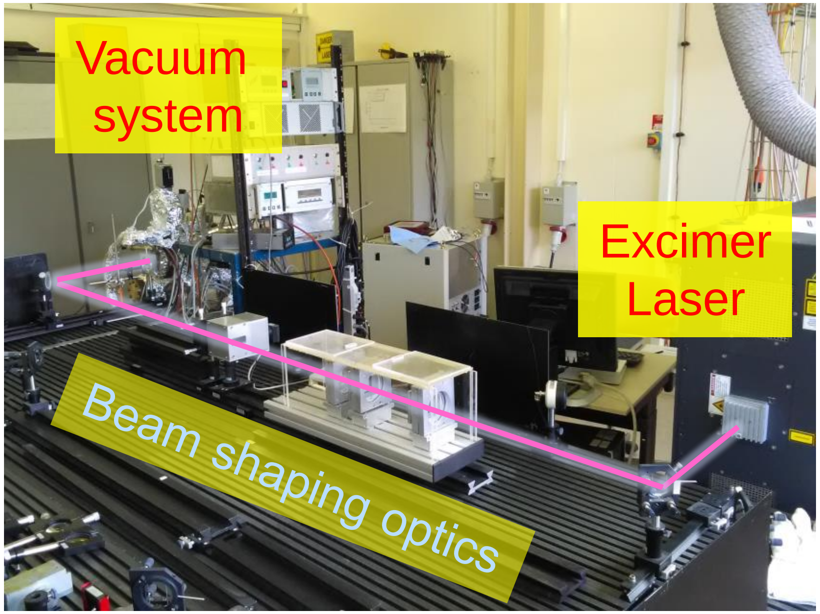
- Cryogenic cooling of samples to 15 K
- SEY, SES, conditioning and XPS analyses at variable temperature
- Optional injection of gases to characterize surface reactions

Pulsed laser ablation

- Ablation of thin films on metallic substrates in UHV by 248 UV laser
- Accumulation of the trapped noble gas in the ablation chamber
- Evacuation of the ablation volume via analysis chamber with a calibrated RGA allows to determine the gas quantity (p•V → number of gas atoms)
- Film thickness × ablated area (2 mm^2) → compute number of ablated atoms
- Gas content = $\frac{\text{\# noble gas atoms}}{\text{\# ablated atoms}}$

Options:

- laser-fatigue tests
- pulsed laser deposition from target



Key parameters:

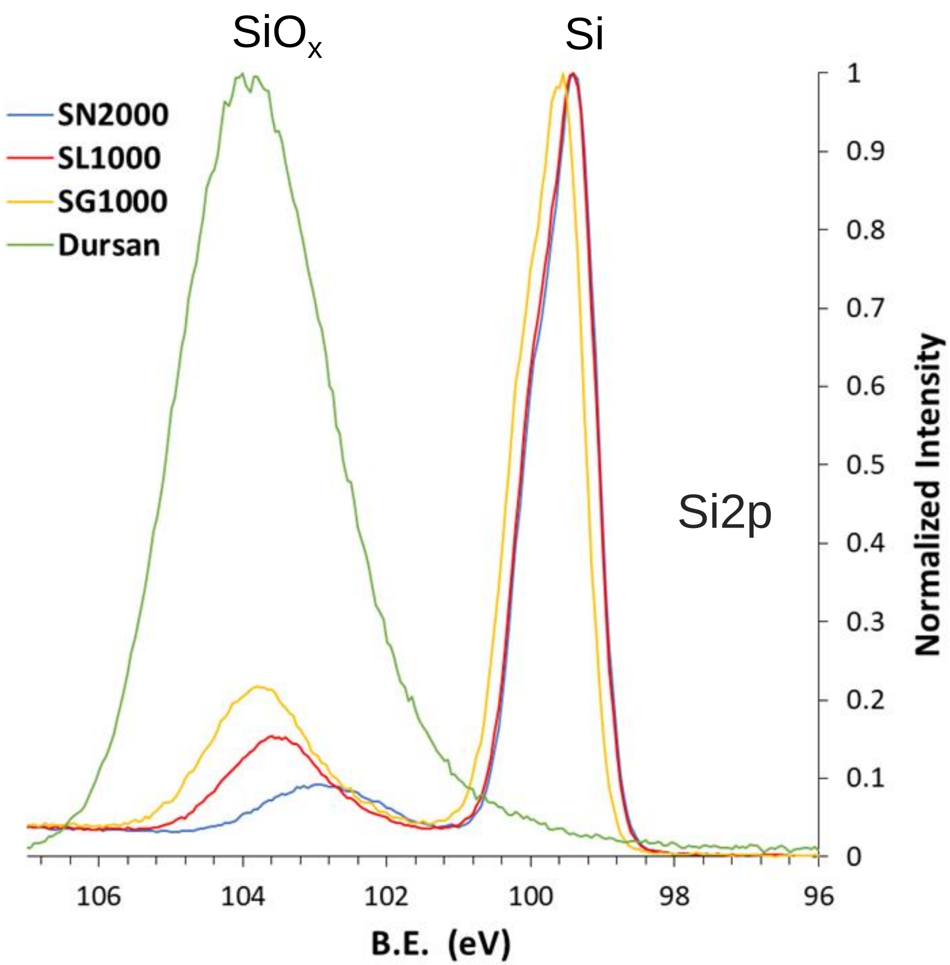
- Possibility to measure all noble gases trapped in thin films up to 5 µm thickness
- Gas content as low as 10 ppm detectable

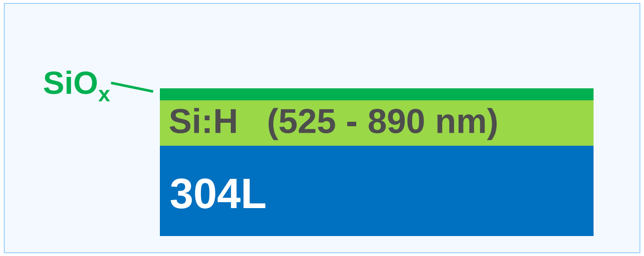
Typical surface analysis tasks at CERN

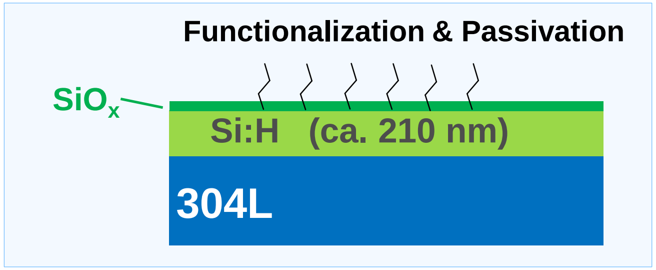
- Contamination analysis after cleaning or material processing
- Thin film composition characterization
- Identification of chemical bonds and adsorbates at surfaces
- SEY qualification of materials and surface processing including electron-induced conditioning tests
- In-situ modification of surfaces & model experiments to develop surface technologies
- Development of new routines for material characterization

Example studies on surface technology

a-Si:H coatings to reduce water outgassing

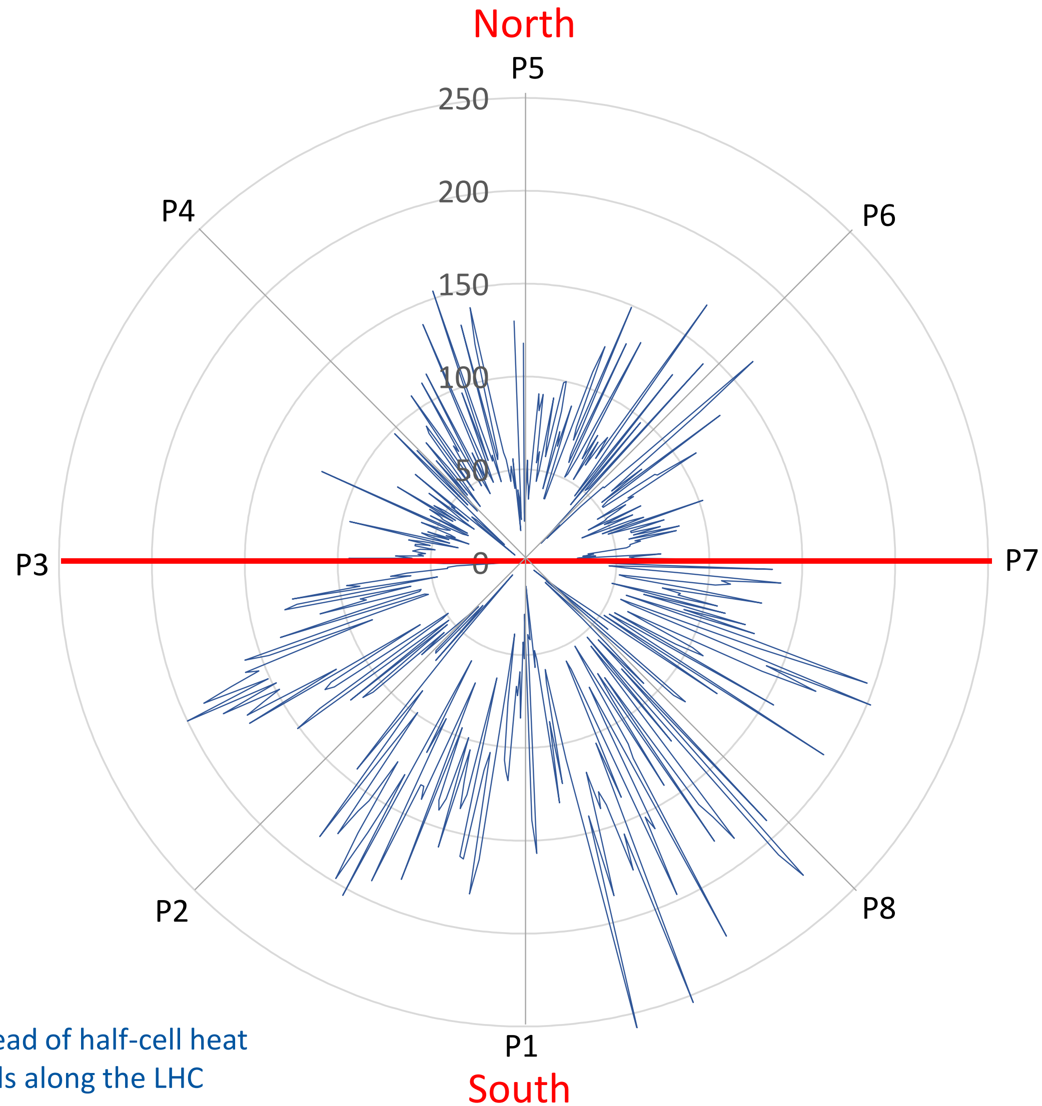






	SN2k	SL1k	SG1k	Dursan
C	7.4	2.2	2.0	17.5
O	16.1	26.9	36.0	47.3
Si	76.5	70.9	61.4	35.2
SiO_x (nm)	0.4	0.7	0.9	-

LHC heat load spread



Spread of half-cell heat loads along the LHC

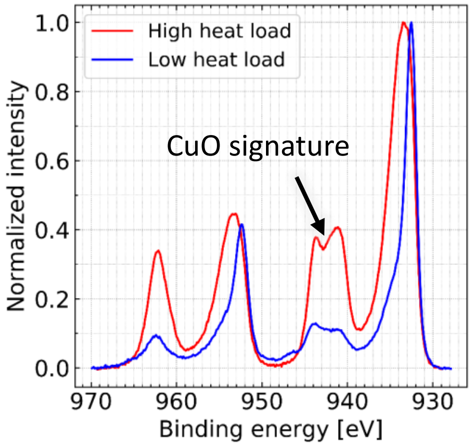
LHC Heat Load Task force:

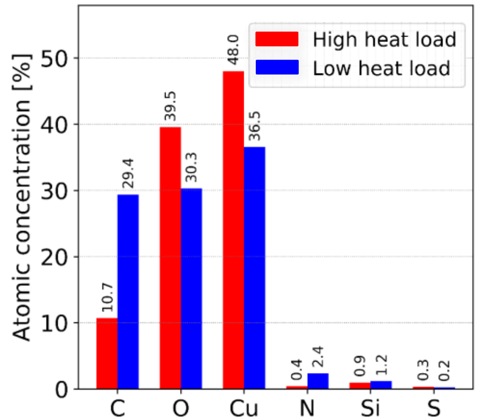
- ✓ Determine possible origins of unequal heat load
- ➢ Development of solutions to mitigate extensive heat load for HL-LHC operation

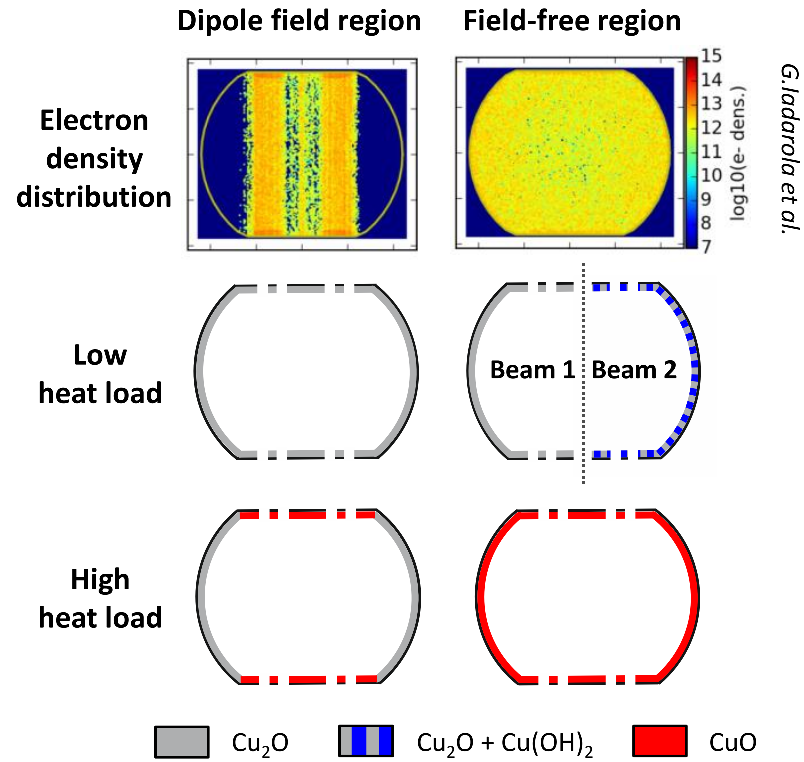
Origin of LHC beam induced heat loads

May-August 2019: extraction of beam screens hosted in one high and one low heat load dipole and characterisation of their surface in the laboratory

→ Surface chemistry (X-ray photoelectron spectroscopy)
→ Secondary Electron Yield
→ Electron conditioning behaviour







In high heat load beam screens

- Presence of **CuO (not native copper oxide)** with a field-related azimuthal distribution
- **Very low amount of carbon** at all azimuths

V. Petit et al., Commun. Phys. 4, 192 (2021)