Surface analysis and properties for CERN's accelerators

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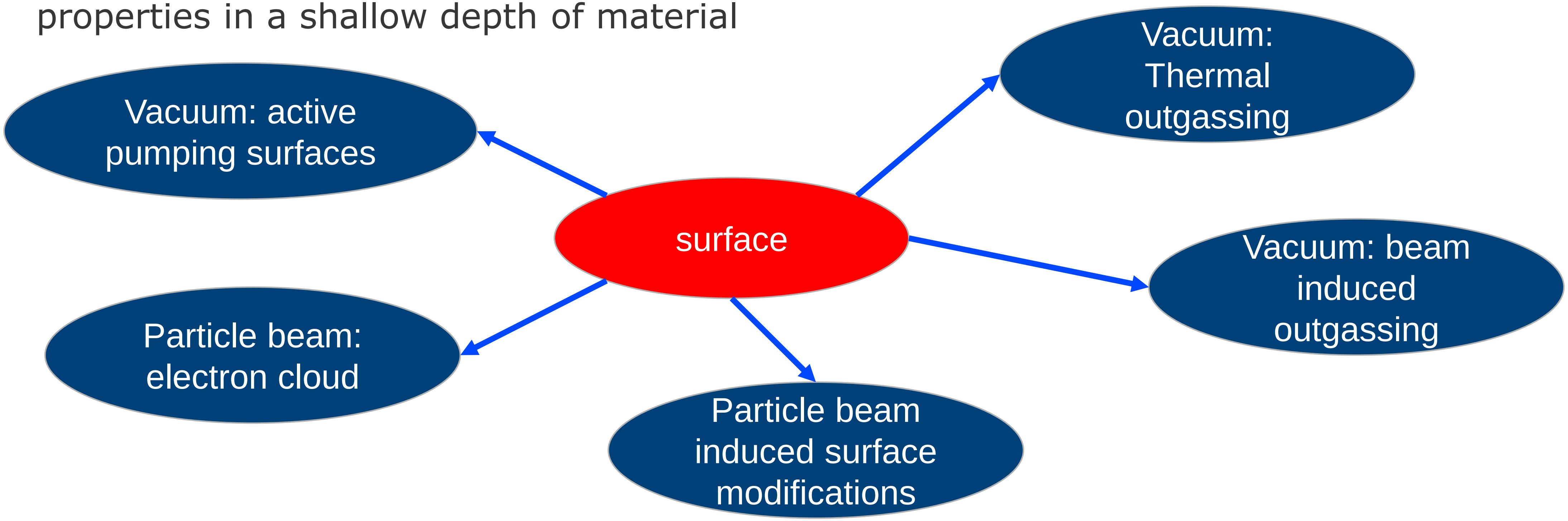
OUTLINE:

Why surface analysis for accelerators?
Surface cleanliness and its assessment

Measurement of surface properties; the case of the Secondary Electorn Yield (SEY).
Laser surface treatment for low SEY

Why surface analysis?

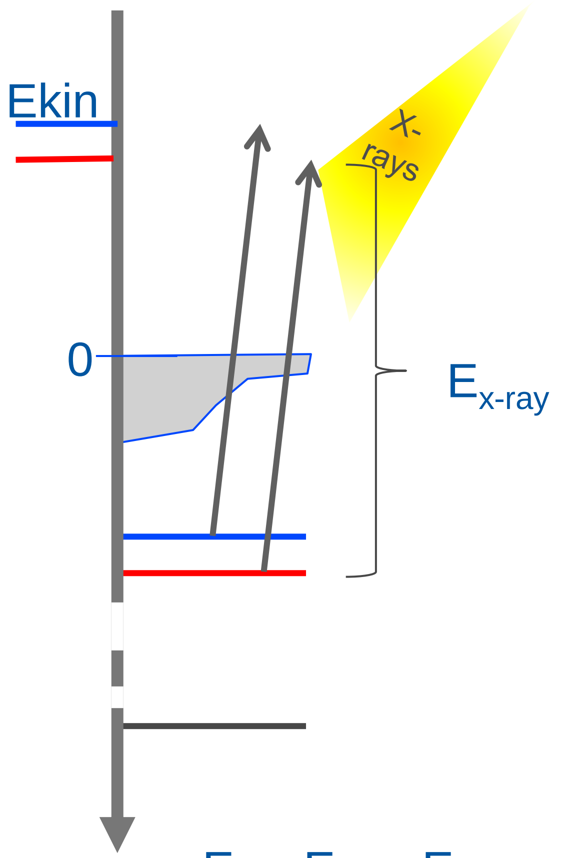
Surface analysis is used to measure the chemical composition and physico-chemical properties in a shallow depth of material



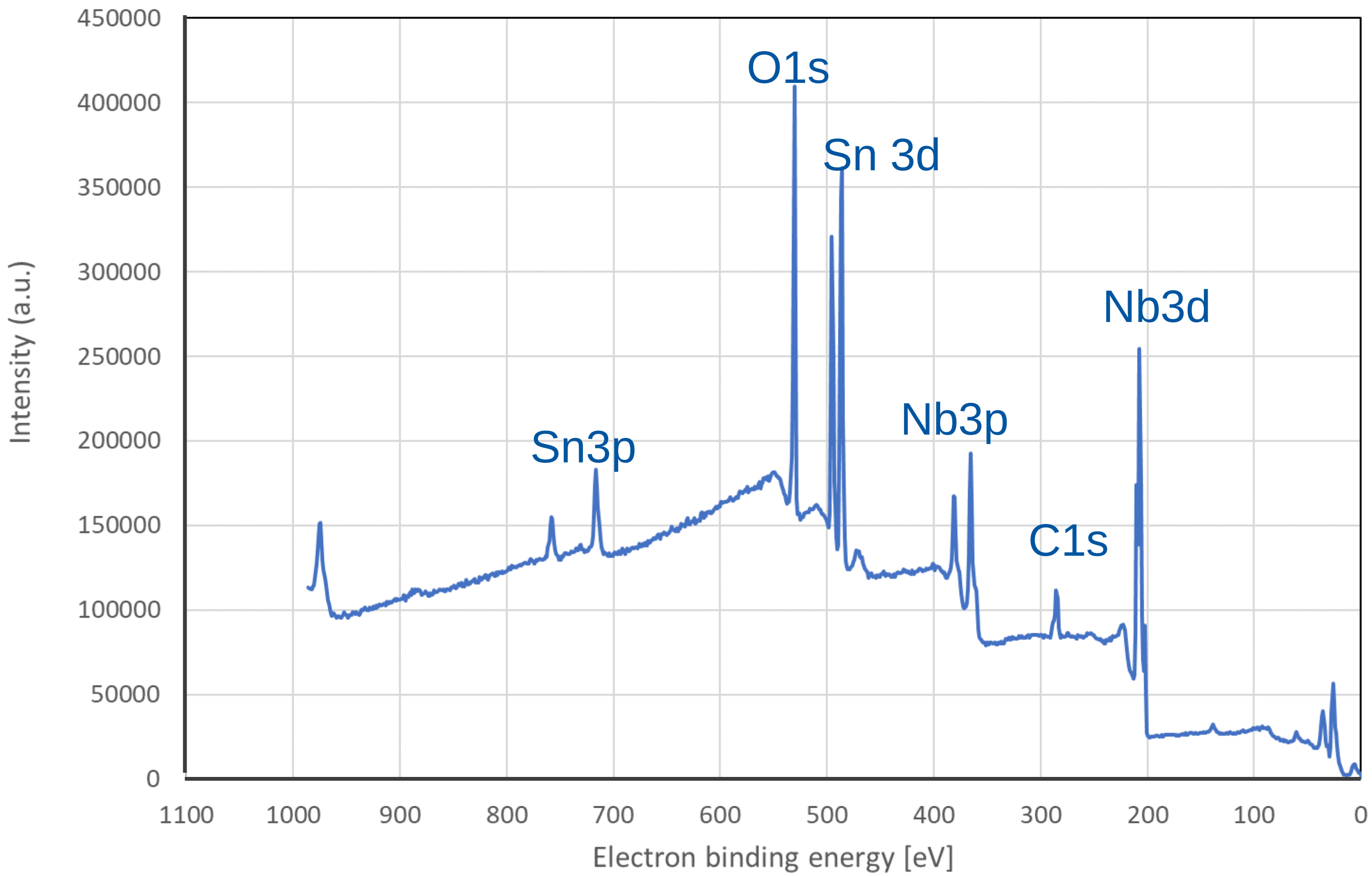
Therefore, we need to characterise the surfaces facing vacuum

Surface analysis by X-ray Photoemission Spectroscopy (XPS)

Electron Binding Energy



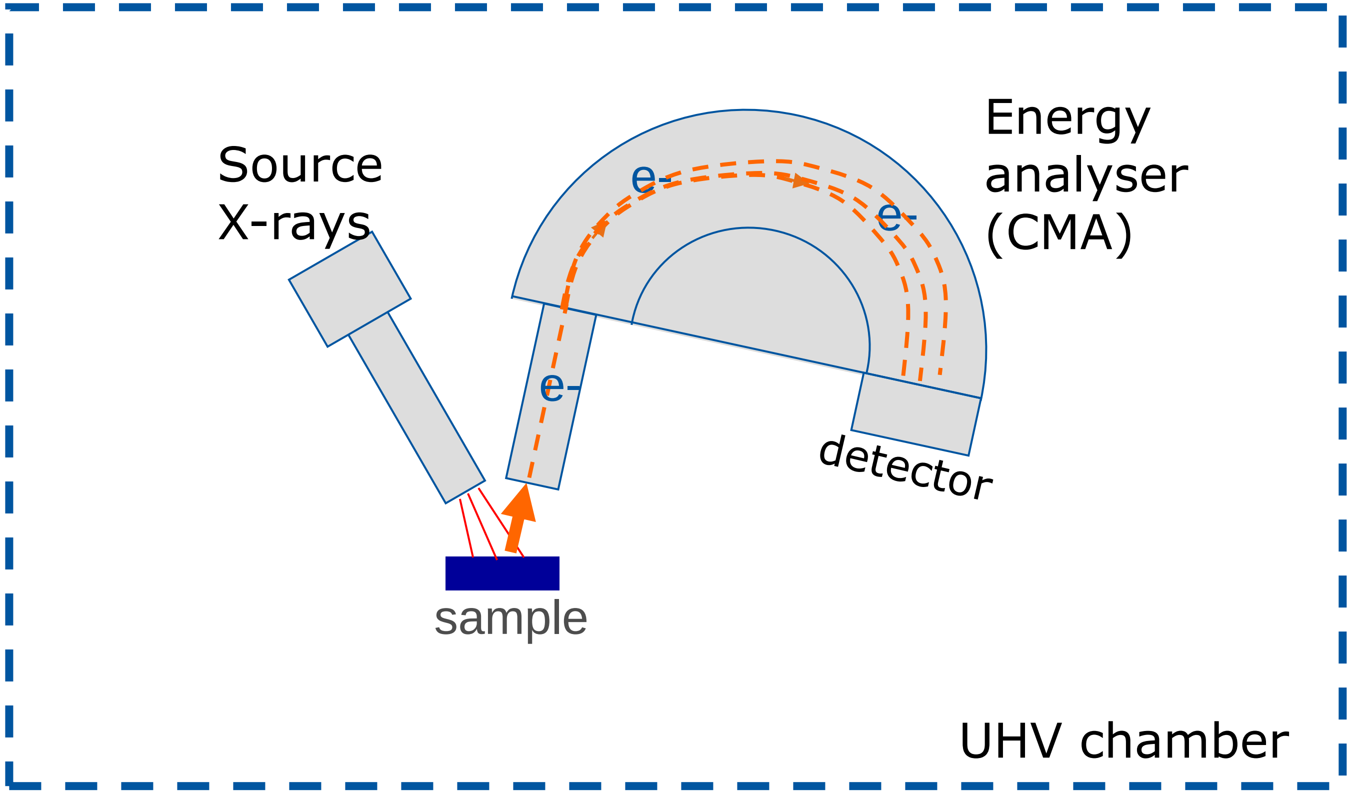
$E_{kin}=E_{x-ray}-E_{binding}-WF$

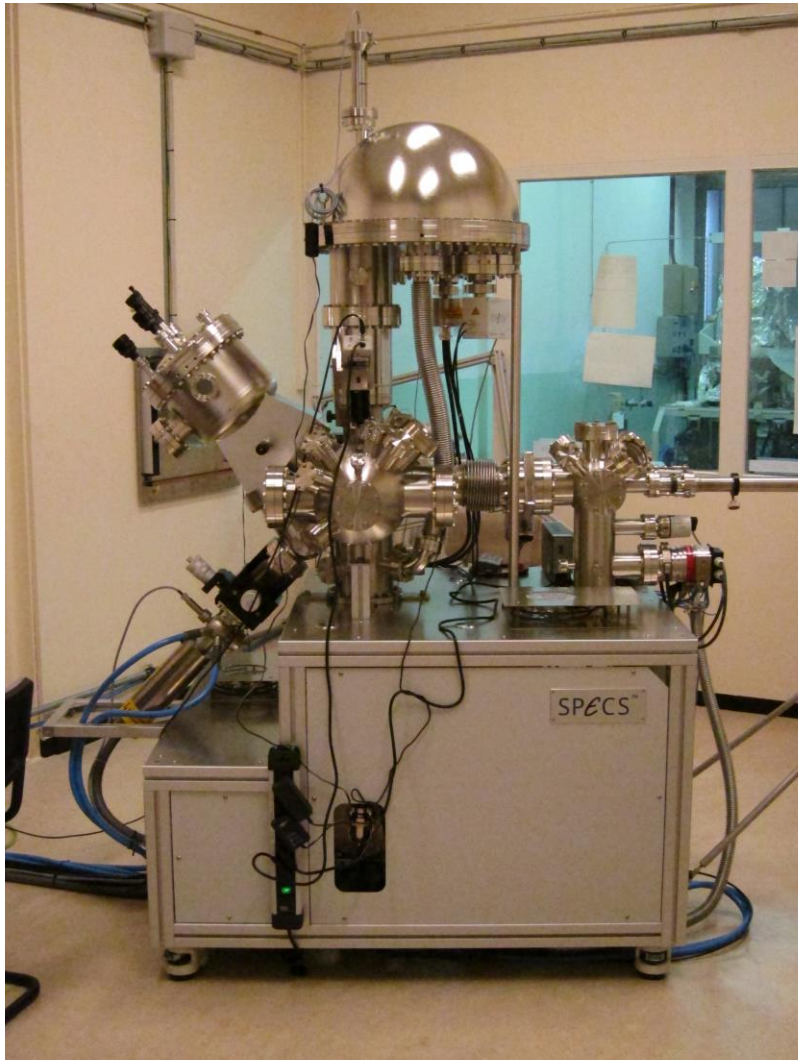


- Signal for different éléments
- Intensity (peak area) proportional to concentration

Surface analysis by X-ray Photoemission Spectroscopy (XPS)

Principle



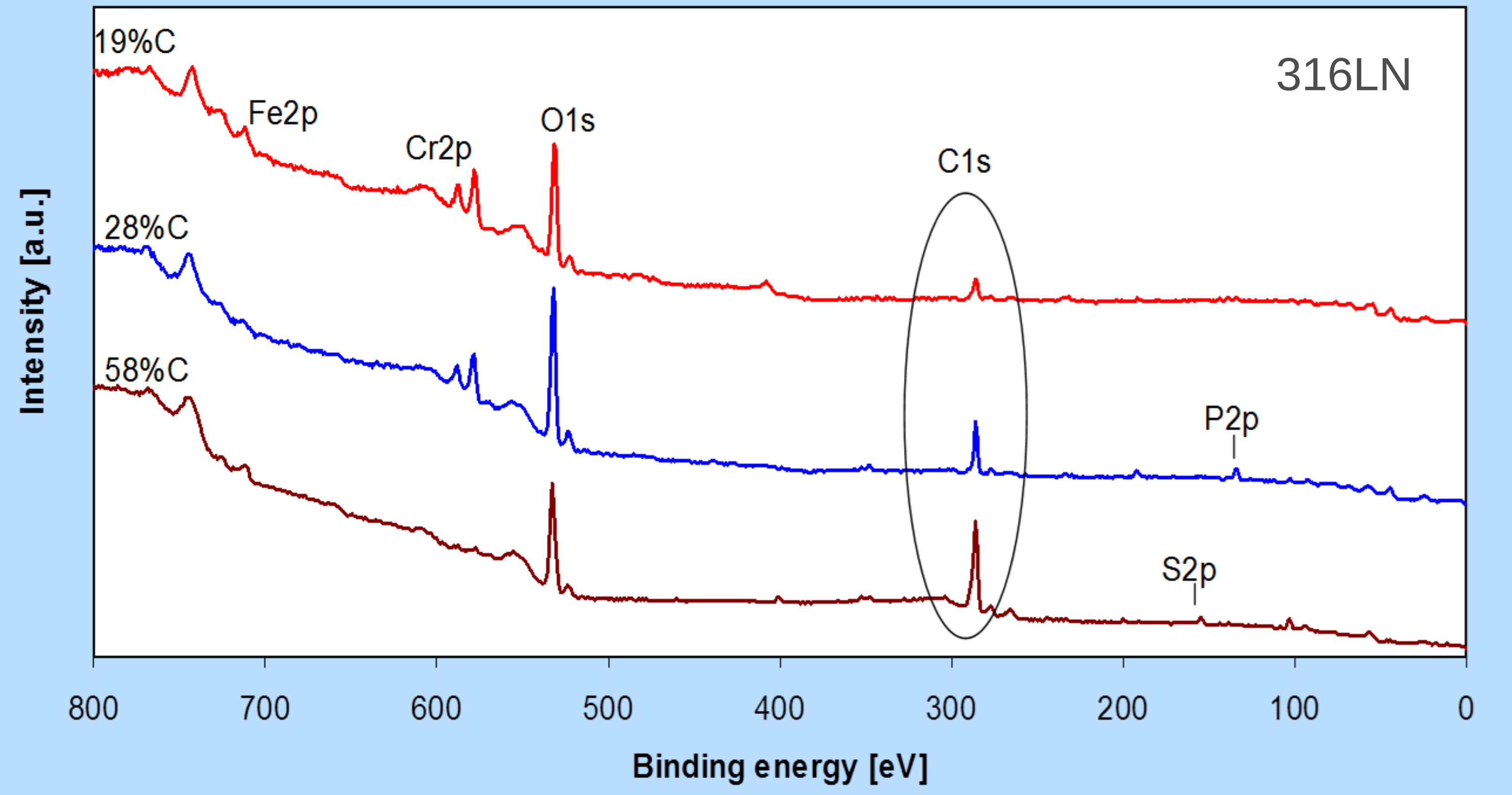


At CERN we have at present 3 instruments, one of them able to measure down to cryogenic temperature (10K), another up to 400C

XPS performance

- Measures chemical composition (elements) in a surface layer, the topmost ~2-5 nm of material
- Detection limit for elements 0.1 at.% or better
- Lateral resolution ~ 200 µm
- Depth profiling (destructive by Ar^+ ion etching) up to ~ 2 µm
- In many cases sensitive to chemical bonds (metal atom bound to oxygen=oxide vs metallic surface etc…)
- Regularly used in industrial process control (semiconductor industry, thin film coatings etc…)
- Analysis generally performed in UHV, but systems exists up to 100 mbar

XPS for cleanliness analysis for UHV applications



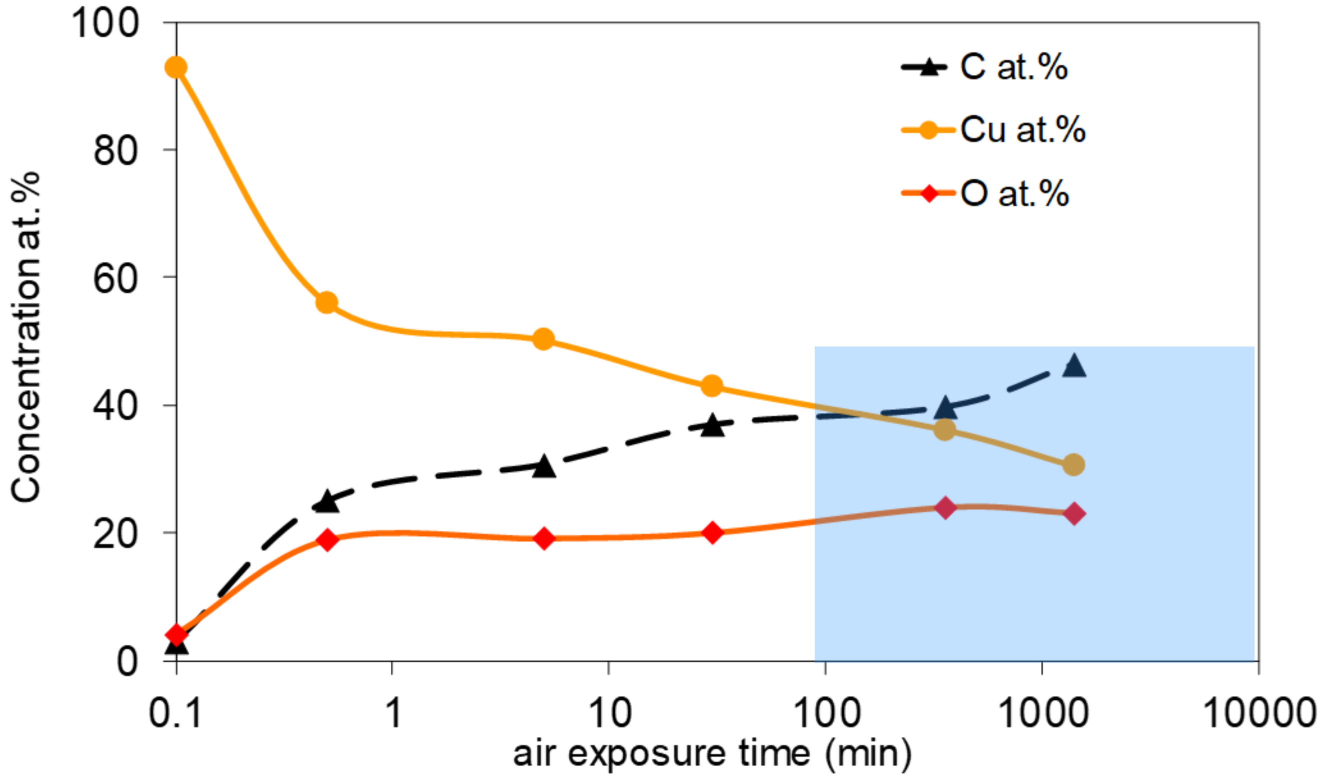
Define criteria for cleanliness:
for instance threshold for **carbon** at%

Sensitivity depends on the substrate, but is well below 1μg/cm²

How fast do we get contamination after cleaning?

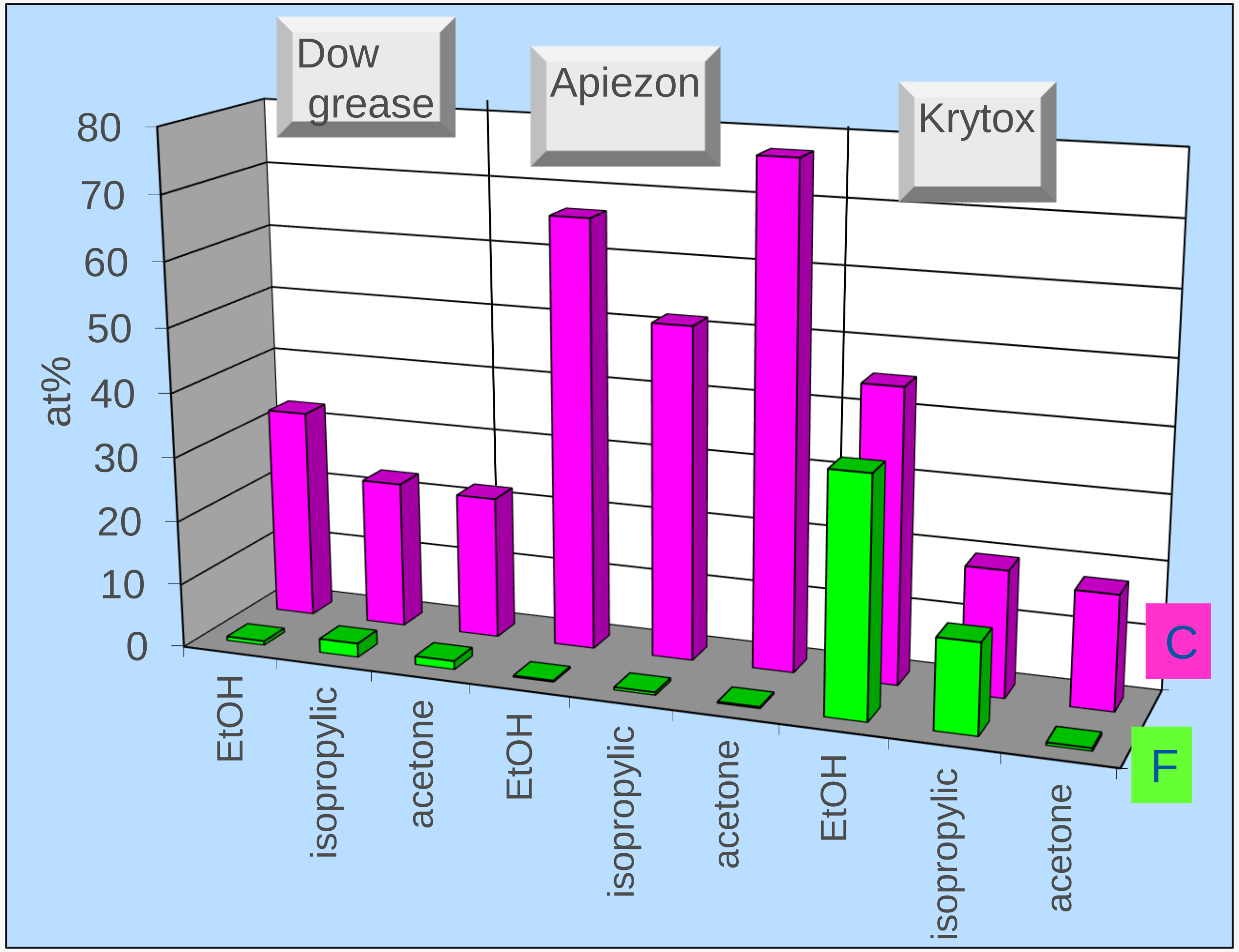
This tells also which cleanliness level we can require/achieve for surfaces exposed to air

Example of copper

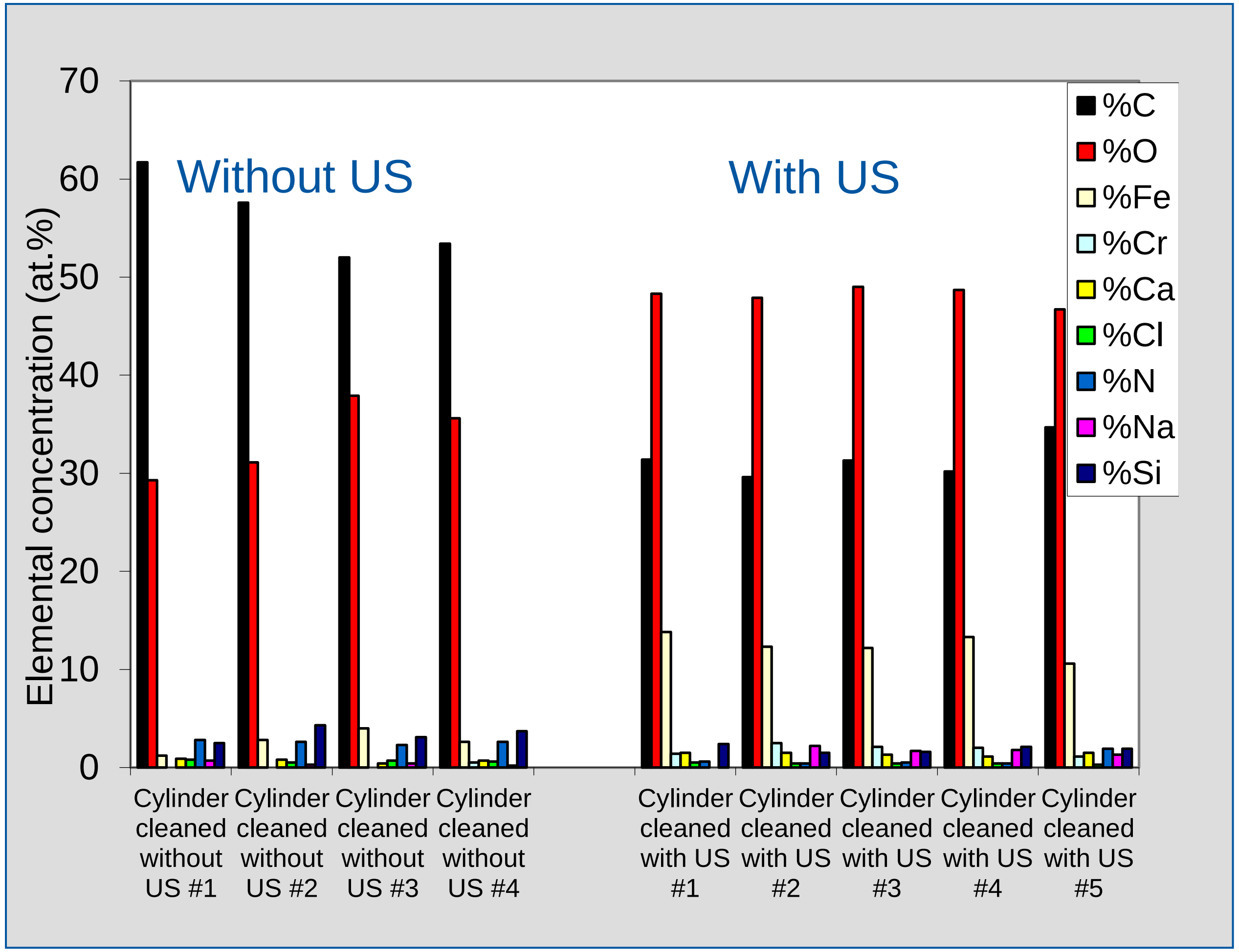


Examples: XPS for cleanliness analysis for UHV applications

Comparison of cleaning greases with different solvents



Comparison of detergent cleaning with and without ultrasonic agitation

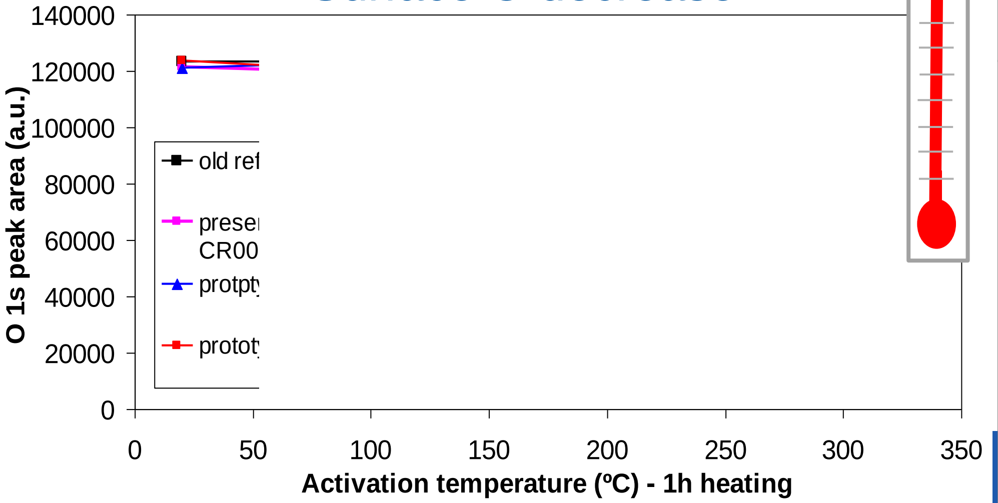


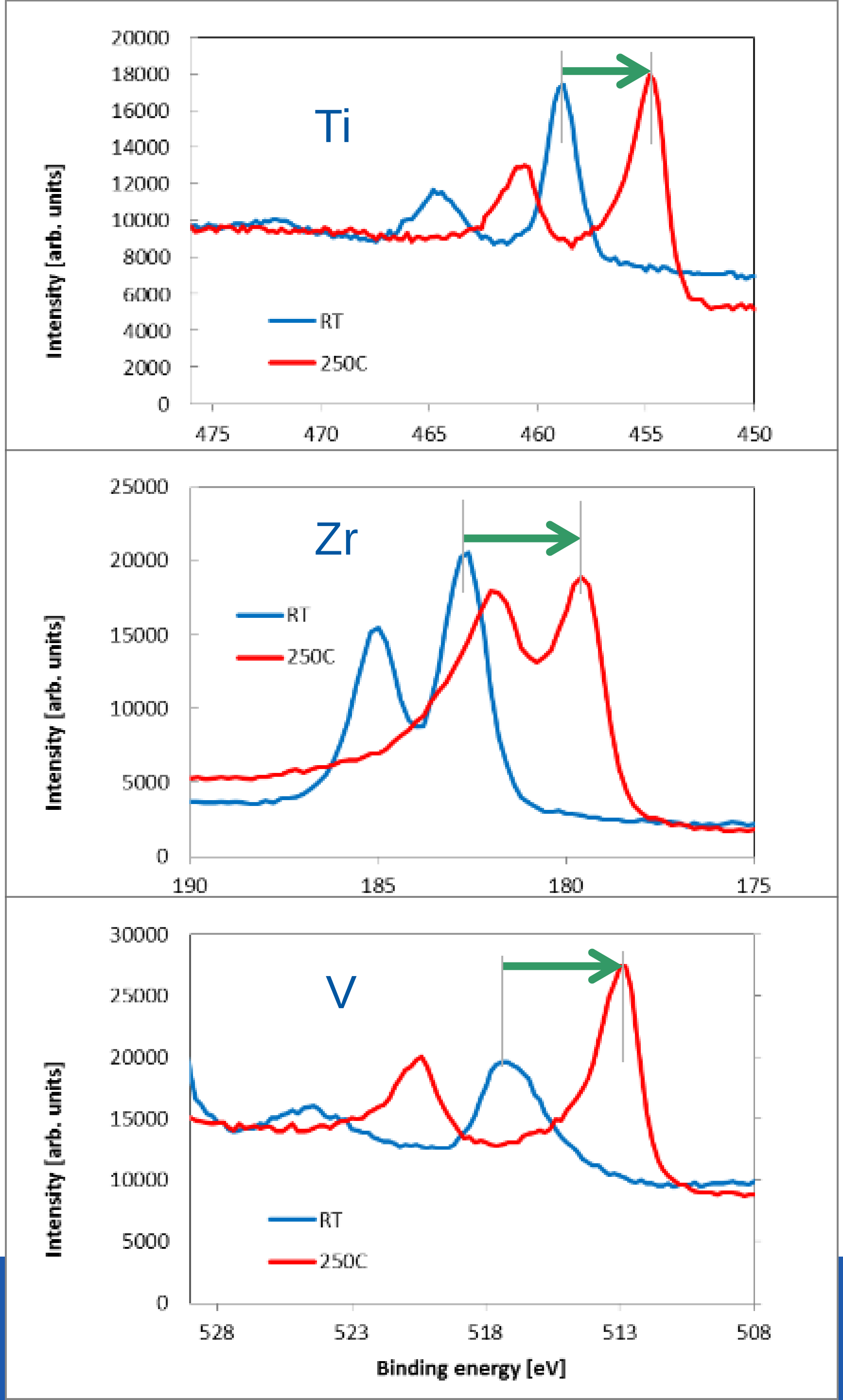
Quality control of thin getter films by XPS:

At CERN we use a TiZrV alloy getter
By XPS we verify:
- the proper ratio of the 3 metals
- the activation kinetics by dissolution in the film of the surface oxygen of the airborne oxide

- ❑ Heating **in-situ**, in UHV, stepwise for 1h at each T
- ❑ Decrease of O on the surface
- ❑ Reduction of the oxides of all the metals: the surface is metallic and reactive

Surface O decrease



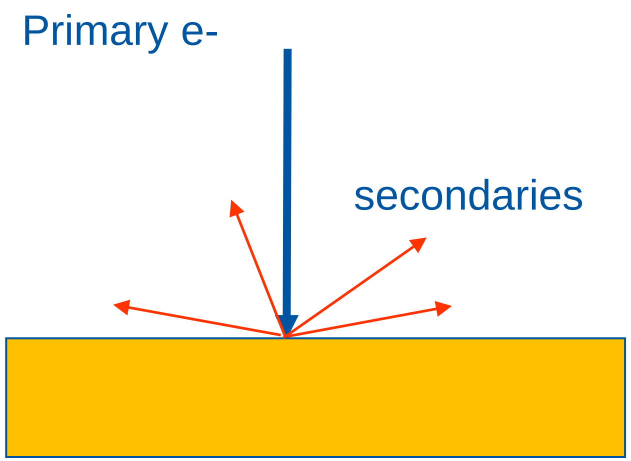


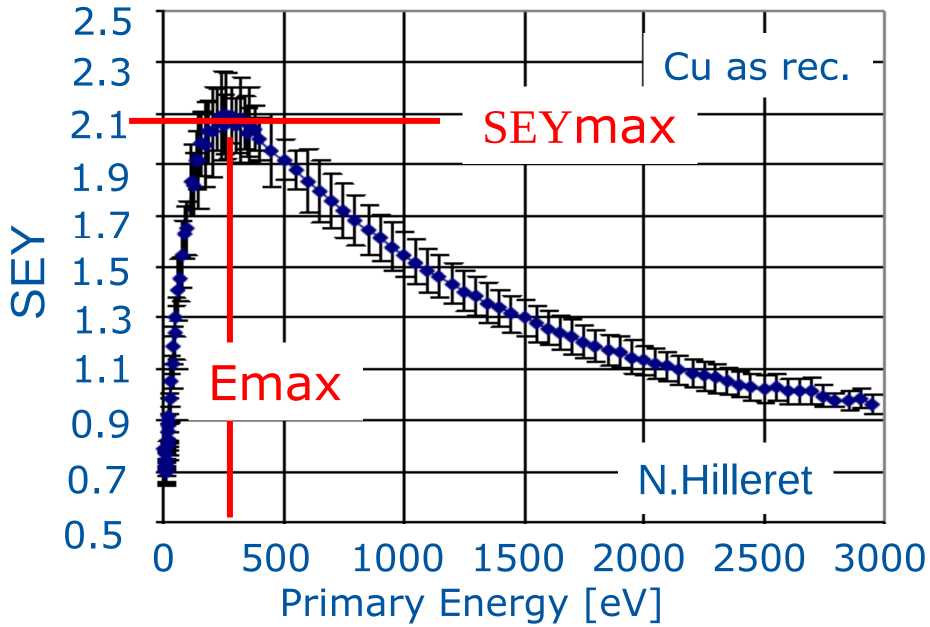
Oxidised

Metalllic

The secondary electron yield: SEY

$$SEY = \frac{\text{number of emitted electrons (secondary)}}{\text{number of impinging electrons (primary)}}$$

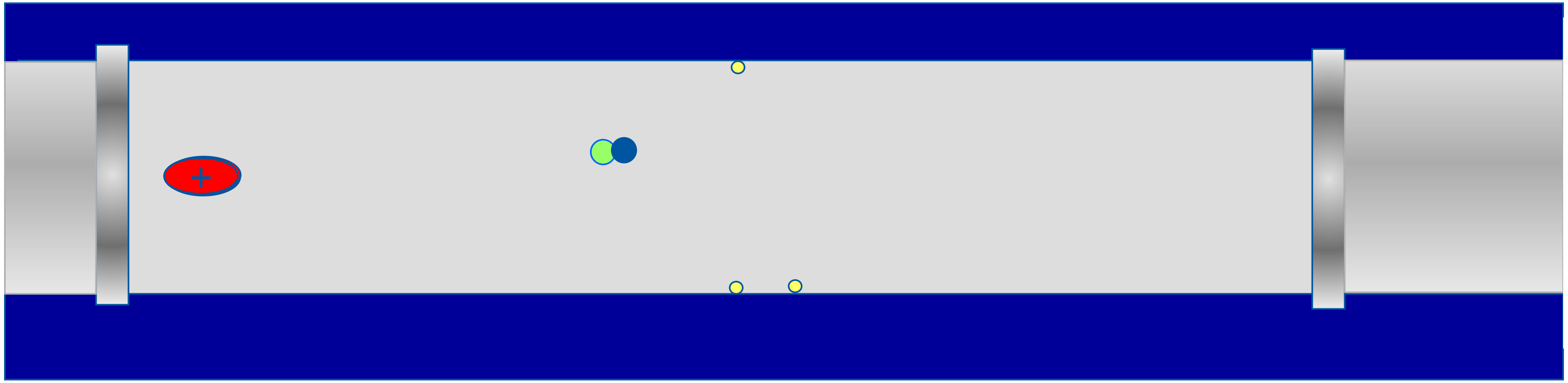




SEYmax = maximum value as a function of **primary** energy
E_{max} = **primary** energy of the maximum

It is the main material parameter influencing electron cloud

What is electron cloud ?



Proton bunch (charge +)

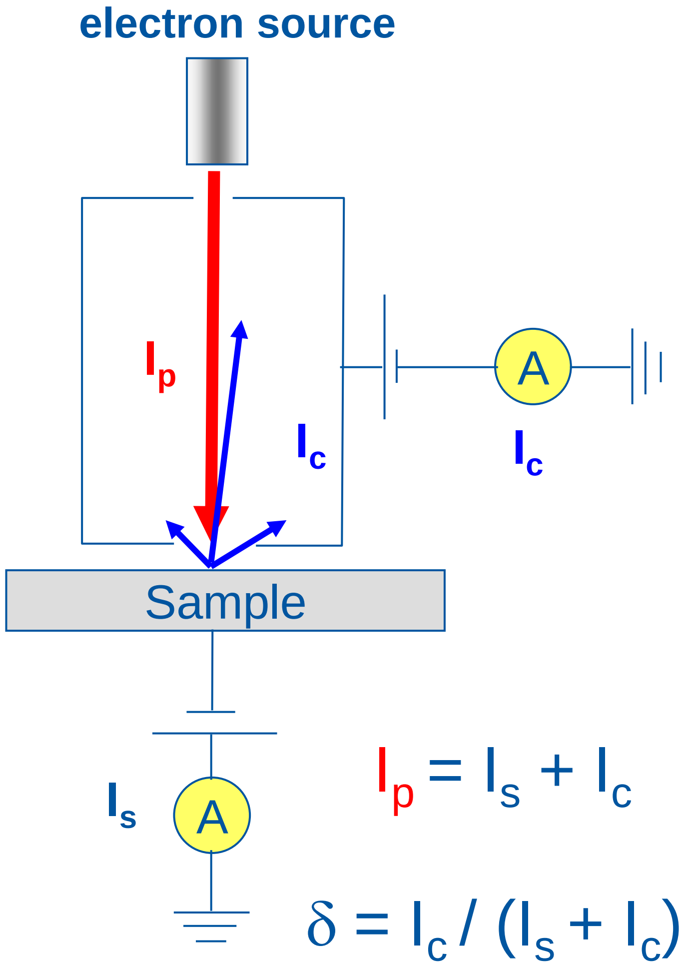
Gas molecule

Electron (charge -)

- The particle beam is perturbed by the electron multiplication
- Cryogenic parts of the accelerator vacuum vessel are submitted to heat load
- The vacuum is deteriorated due to electron stimulated desorption (noise in the experiments, radiation…..)

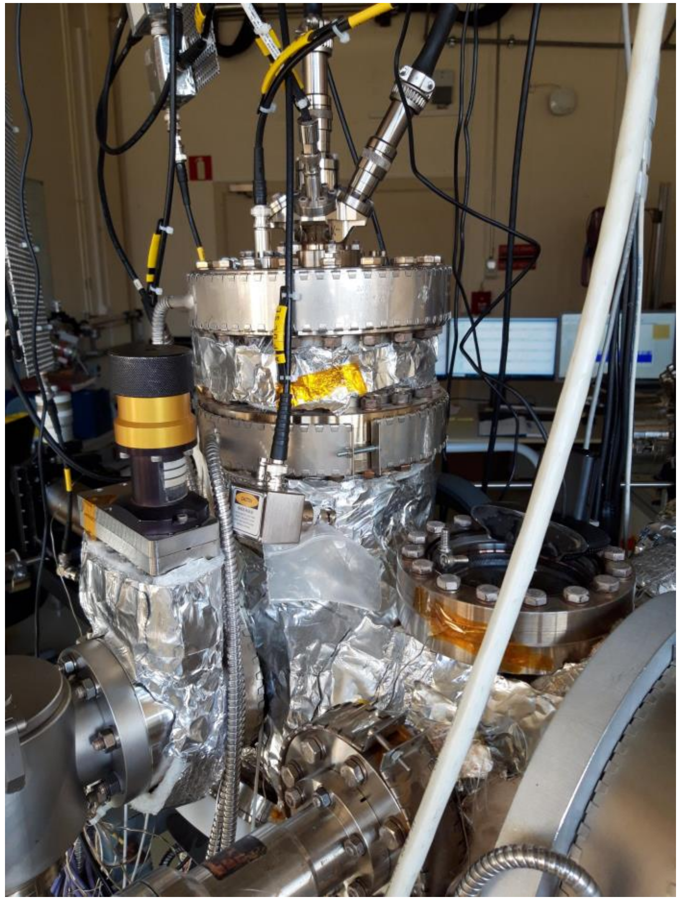
e-cloud is suppressed by a sufficiently low secondary electron yield of the walls, close to 1.

Measurement of Secondary Electron Yield on materials in the lab



$I_p = I_s + I_c$

$\delta = I_c / (I_s + I_c)$

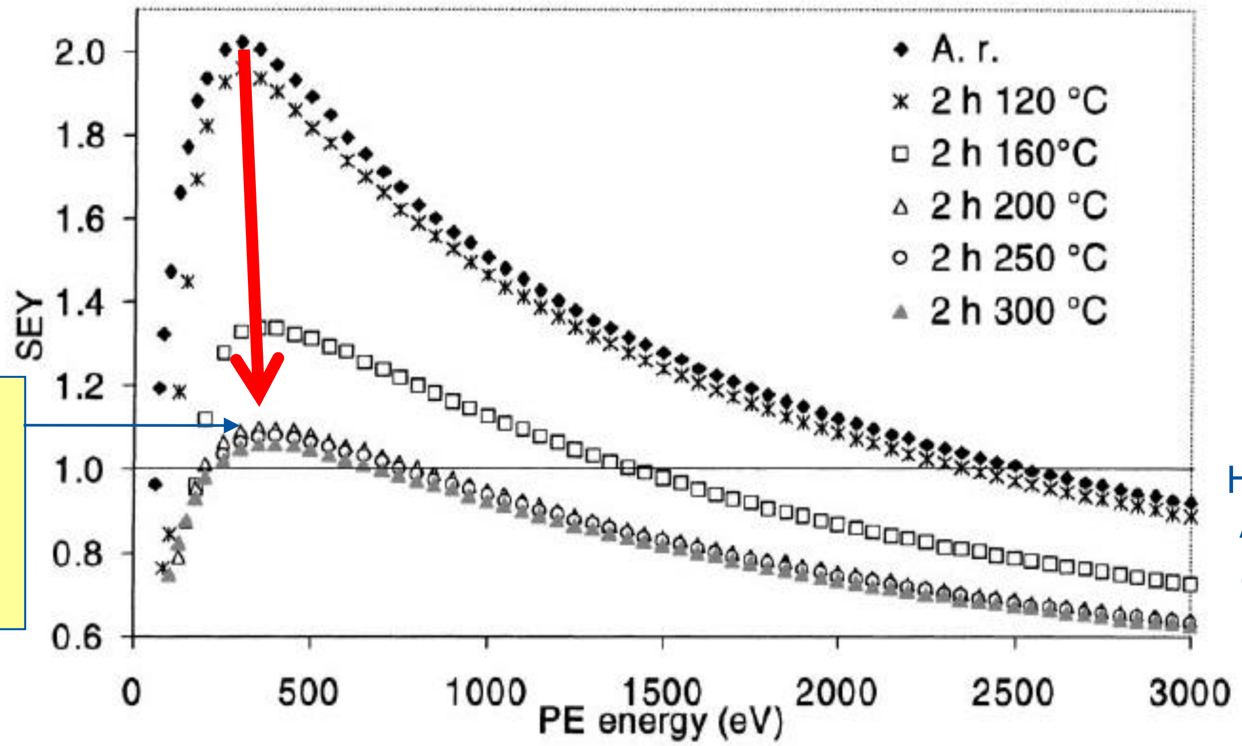


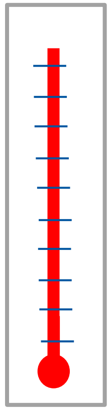
- Measurements done at low dose (below 10^{-6} C/mm^2) to avoid conditioning of the surface
- 3 systems at CERN, coupled with XPS in the same UHV chamber

SEY of Non Evaporable Getter (NEG) coatings

After air exposure and thermal activation in vacuum TiZrV NEG thin films can provide a surface with sufficiently low SEYmax **:**

activate at 200C 2h measure at RT



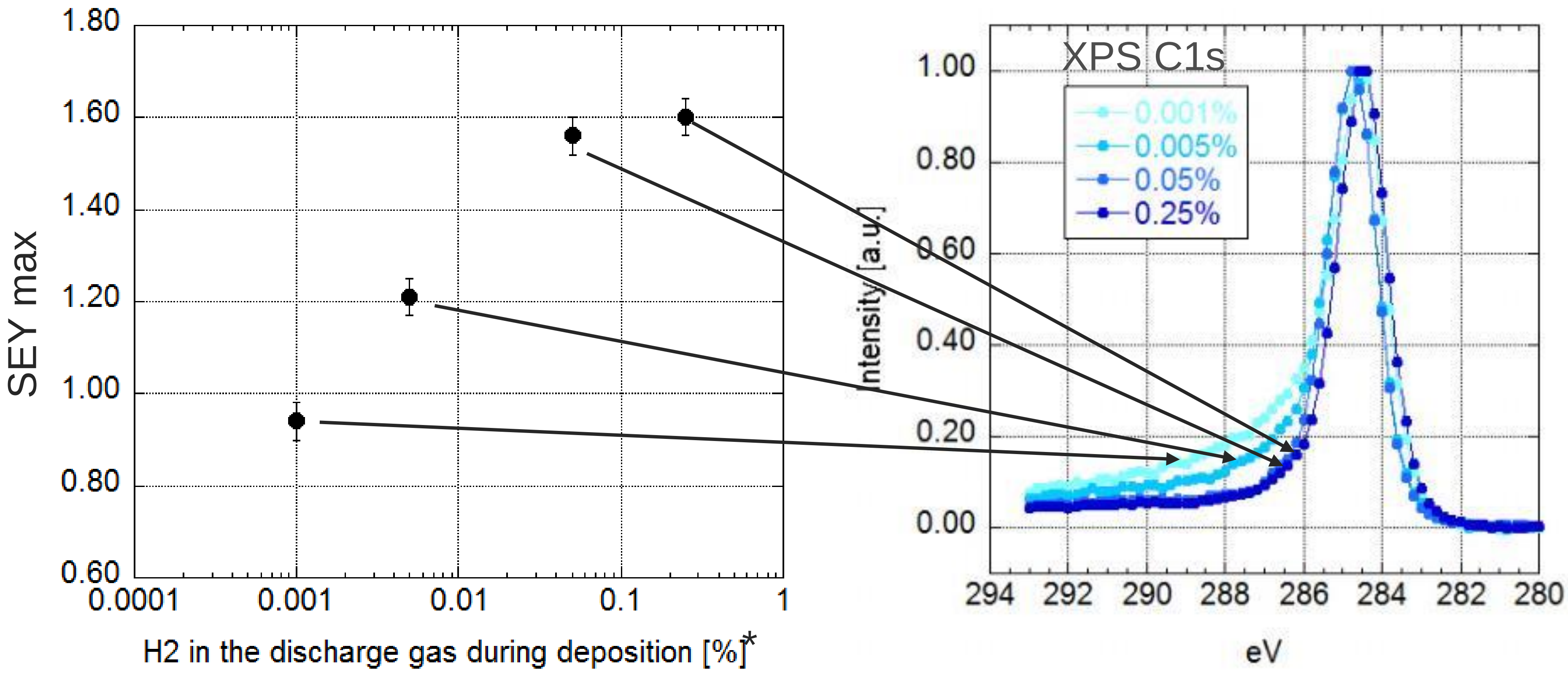


Henrist et al. Appl.Surf.Sci, 2001

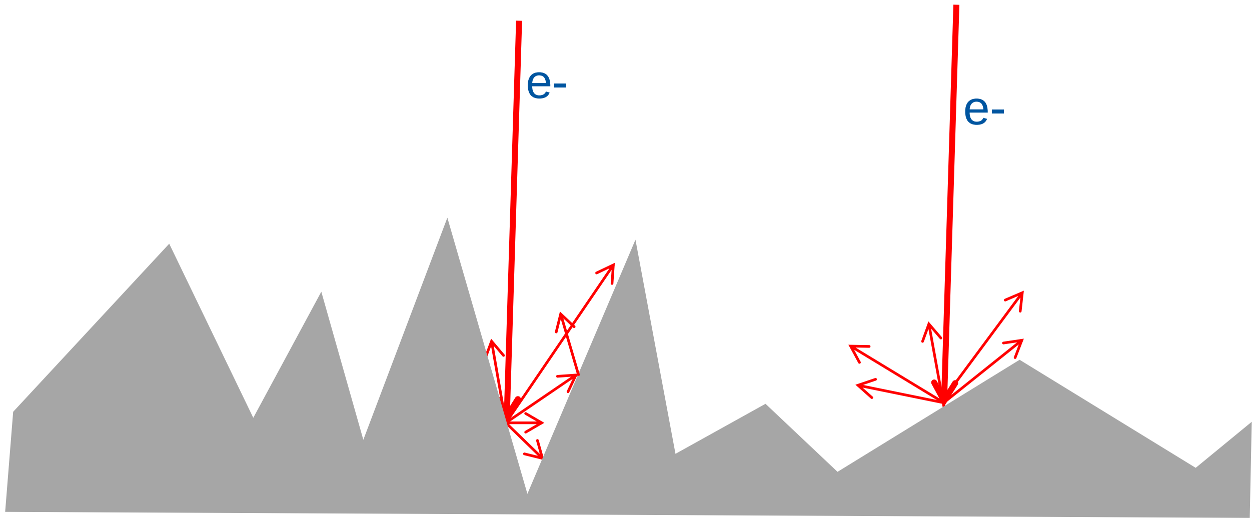
-Thermal activation is necessary: 2h at 200C or 24h at 180C
-Applied in all Long Straight Sections (RT) of LHC and in the experimental chambers, 7 Km of coating

Amorphous carbon coatings for low SEY

- carbon coatings were developed at CERN to obtain low SEY surfaces
- some issues of reproductibility of the SEY lead us to study the effect of H contant
- we could correlate the presence of H_2, as contaminant, in the process gas with a change in SEY and C1s XPS spectral shape



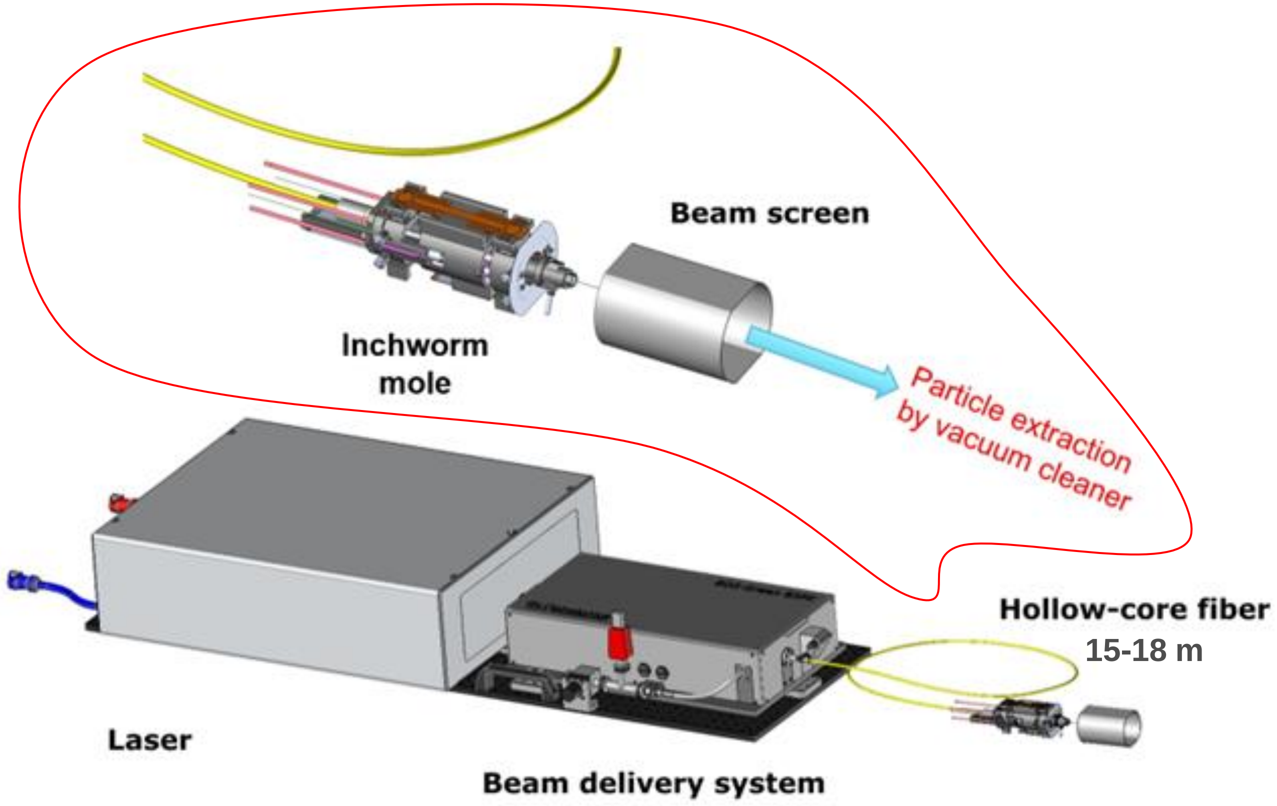
Reduction of the Secondary Electron Yield by roughness



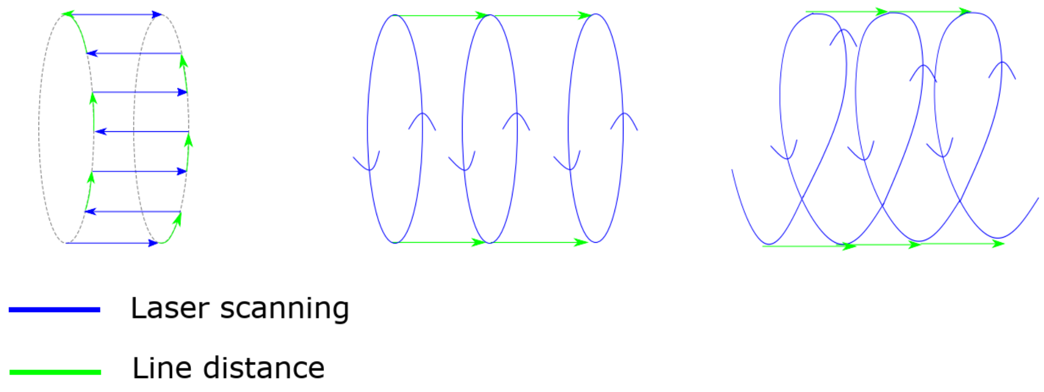
A structure with high aspect ratio hinders the emission of electrons

Laser Surface Structuring to reduce the Secondary Electron Yield

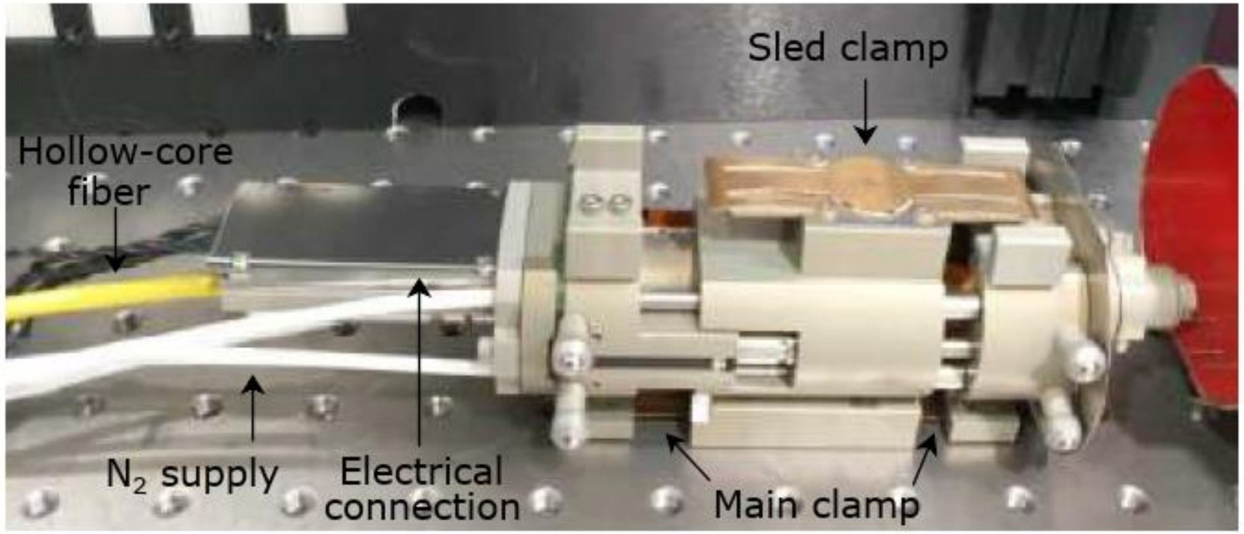
- focussed high power ultra-short-pulsed laser modifies the surface properties of the material
- pulsed laser source (1030 nm, 20W, 1 ps, 500 kHz), (532 nm, 25W, 10 ps , 100KHz)
- 30 mm/s line treatment, 50 μm pitch
- Effective treatment speed of the order of 100 s/cm^2

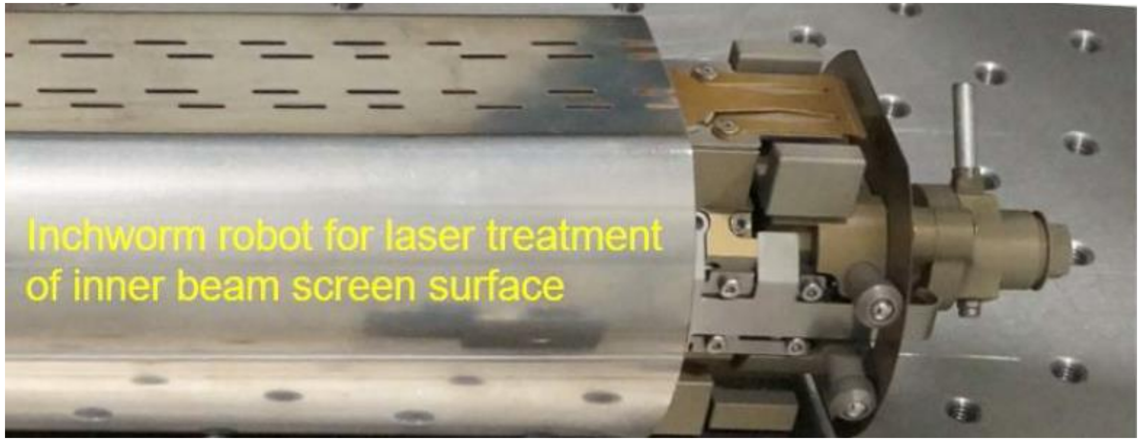


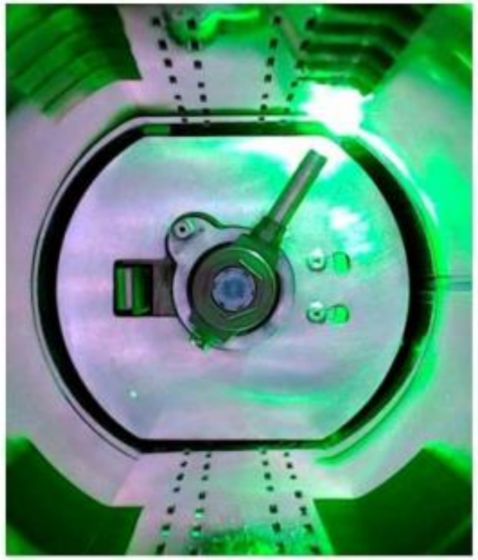
Scanning modes on the surface



Treat tubes and beamscreens: crawling robot

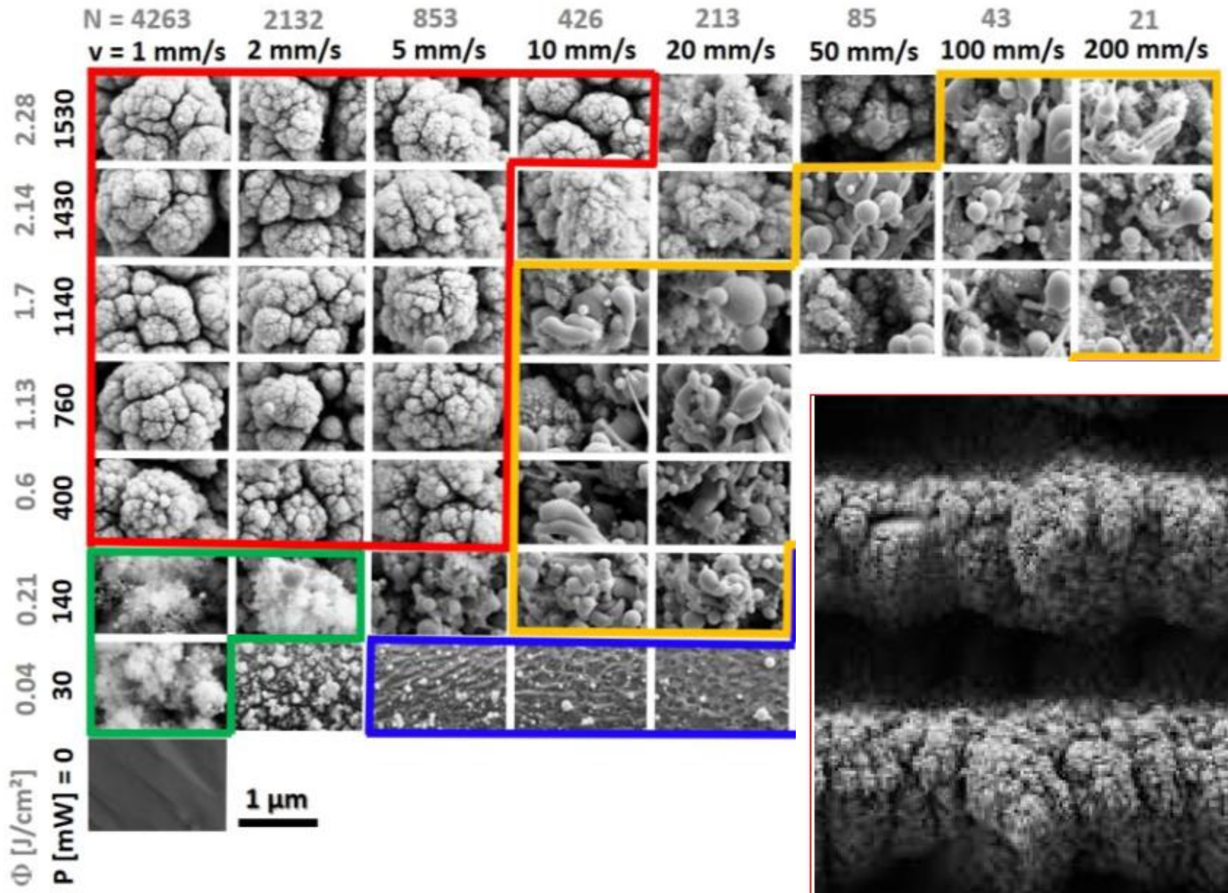


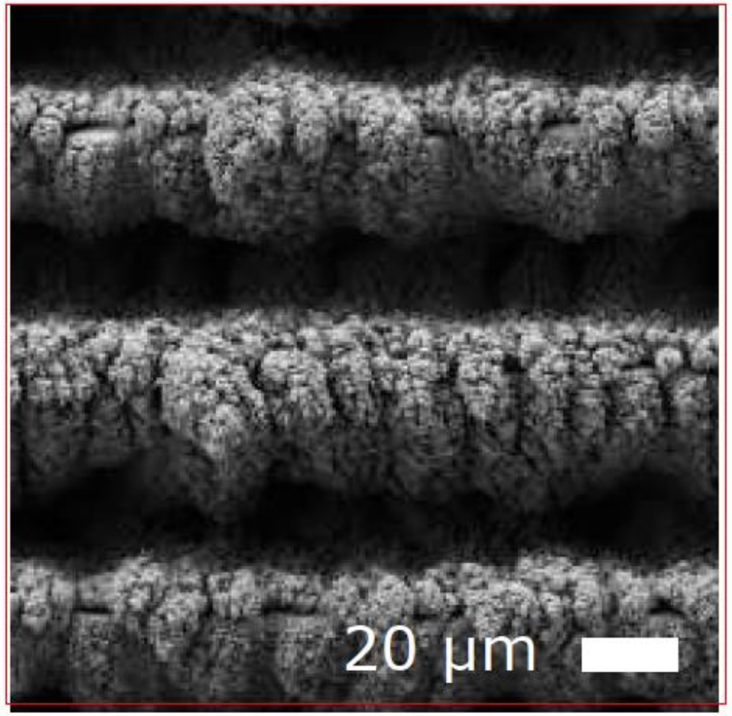


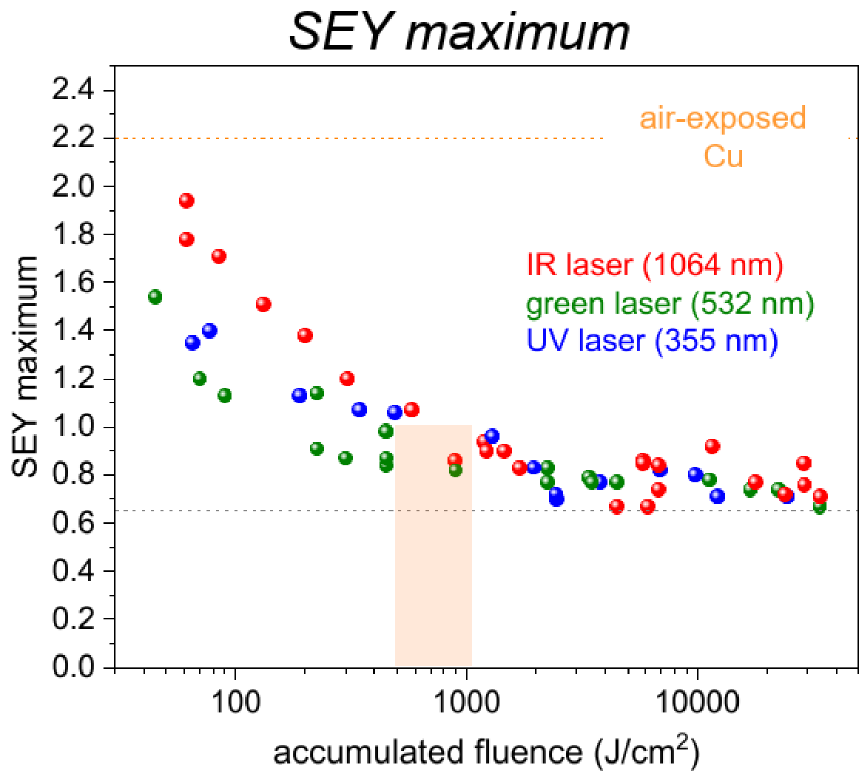


50 mm

Laser Surface Structuring to reduce Secondary Electron Yield







Thank you for your attention

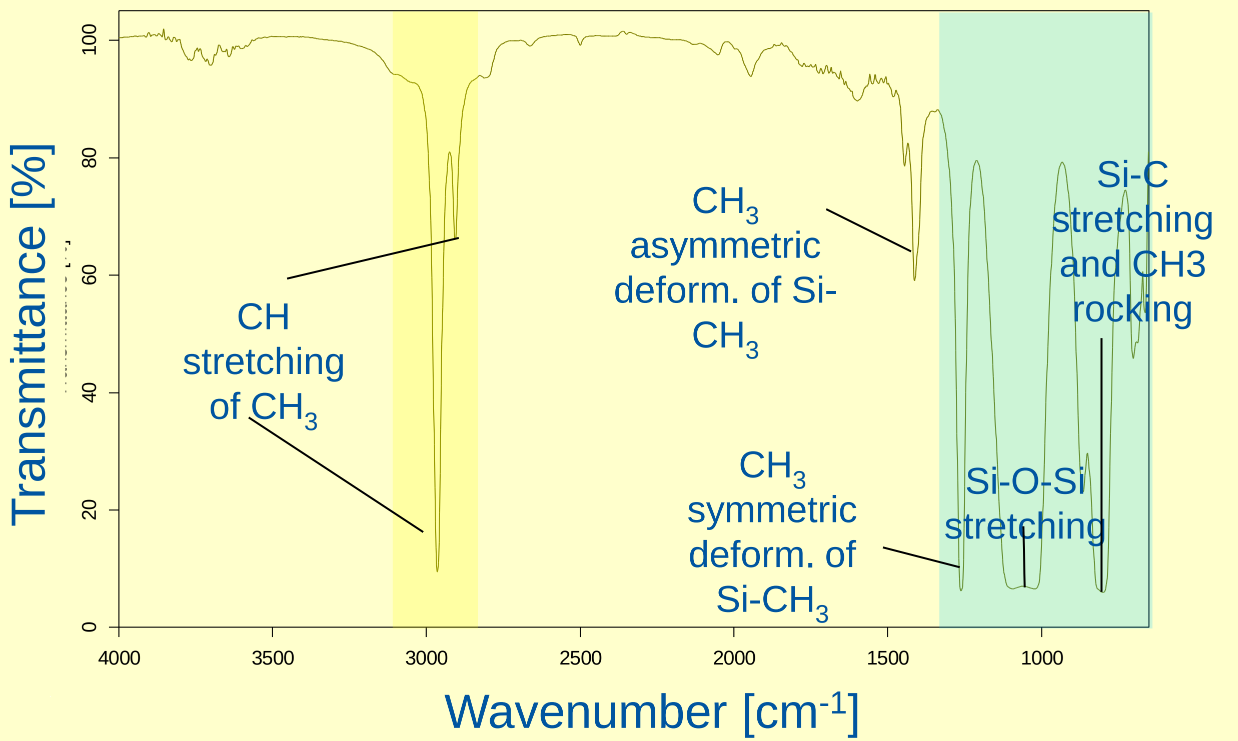
Special case: silicones contamination detection by Fourier Transformed Infrared Spectroscopy

Problem: XPS does not manage to distinguish silicones and silicates (present in detergents to cope with water hardness)

Solution: use FTIR

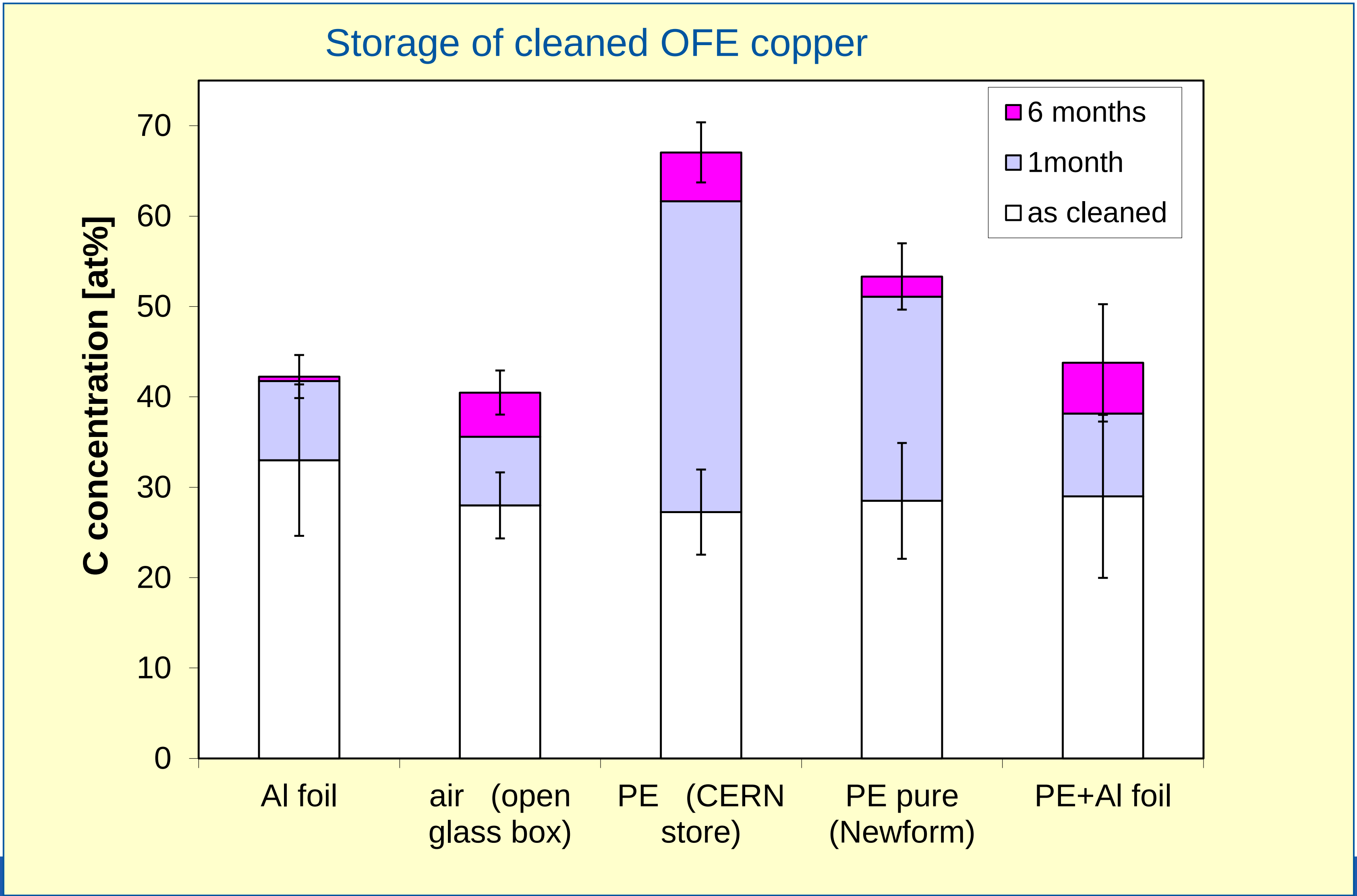
❖ Dissolve the contamination in a fixed quantity of solvent (hexane) per surface unit

❖Analyse a drop of solvent on an IR transparent window (ZnS) and calculate back the surface coverage of silicones

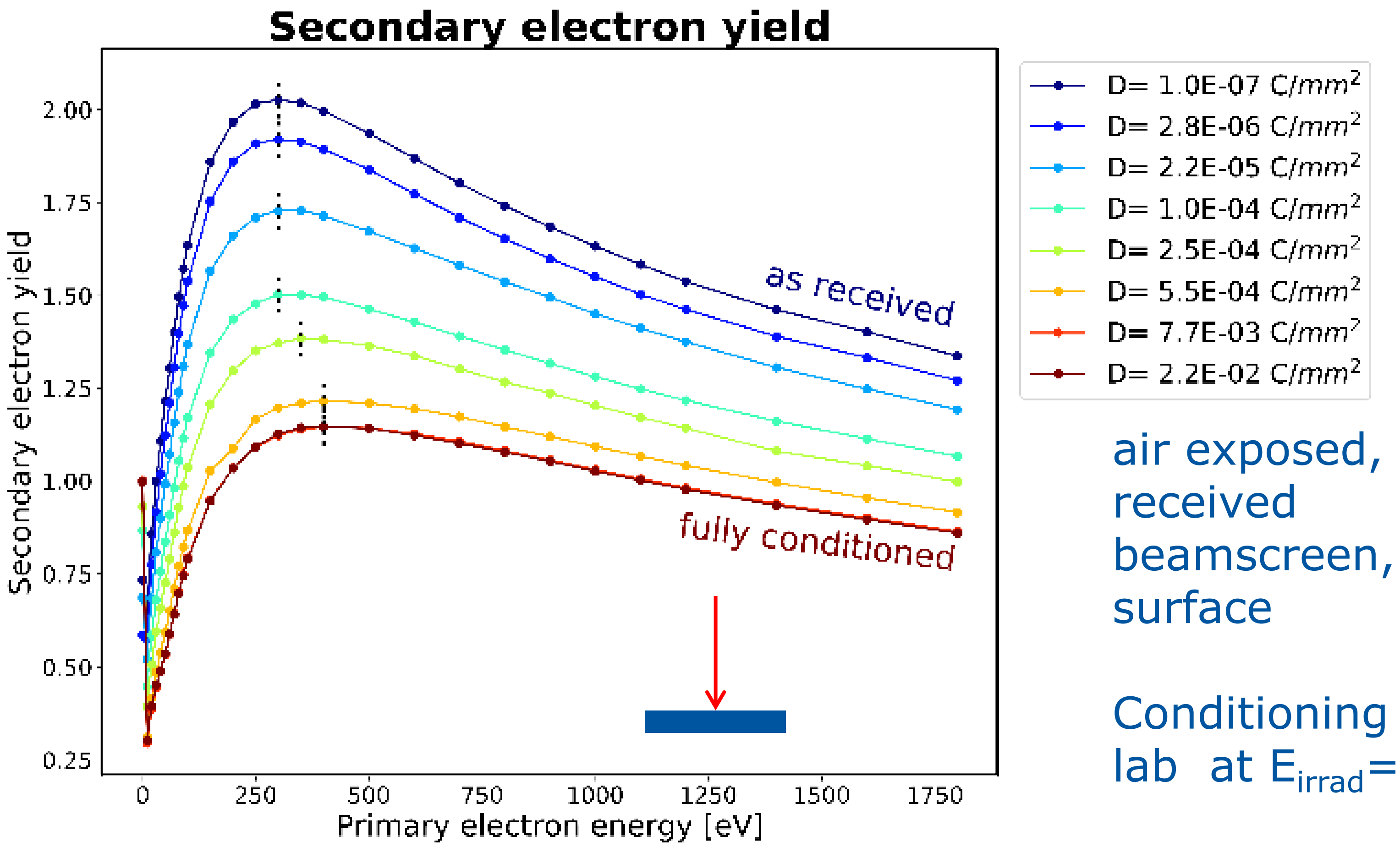


S.Ilie, C.Petitjean, C.Dias-Soares

Effect of storage in different packaging after cleaning



SEY(Ep) of LHC beamscreen copper at RT

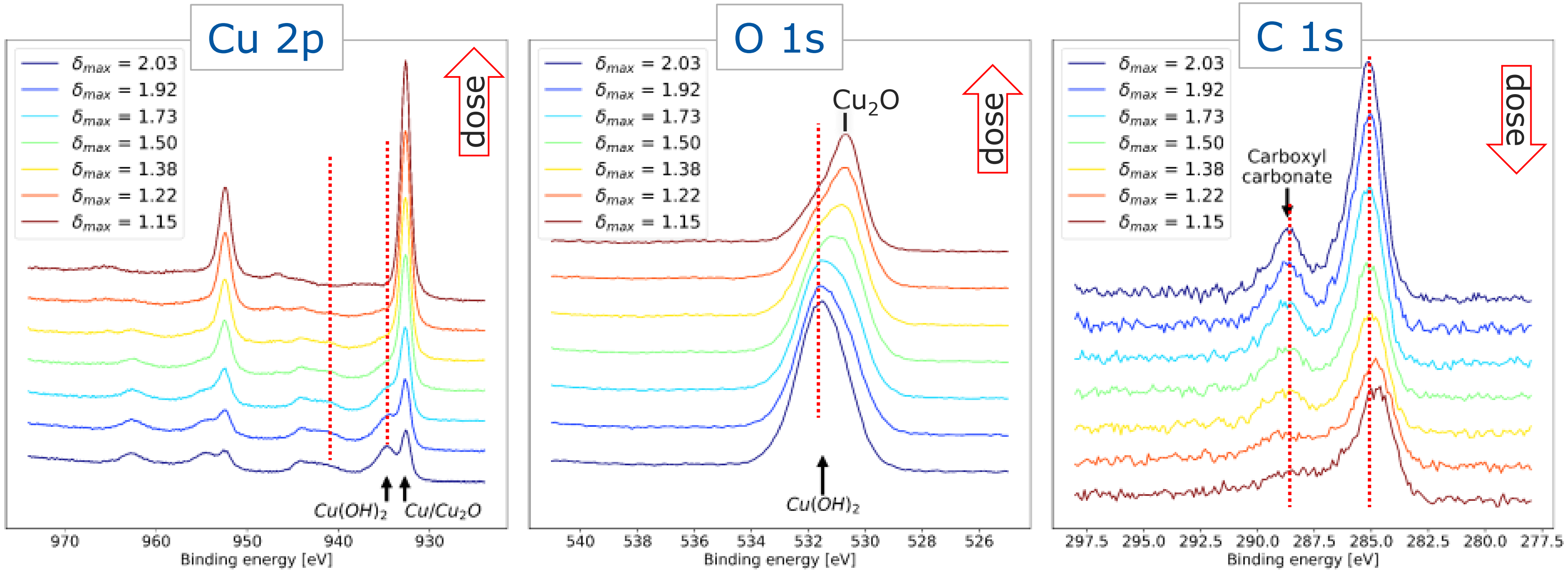


air exposed, as-received beamscreen, copper surface

Conditioning in the lab at E_{irrad}= 250eV

What happens to the surface?

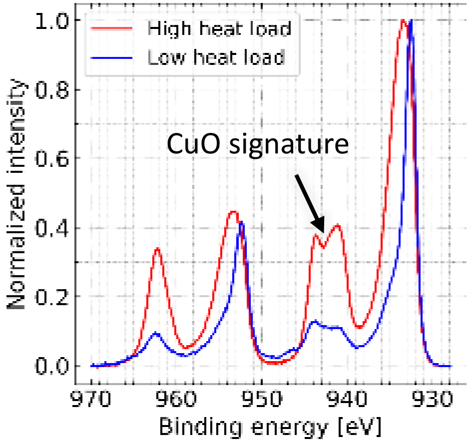
Stepwise monitoring by XPS during irradiation

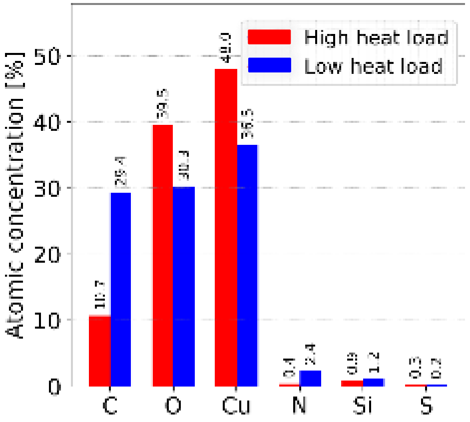


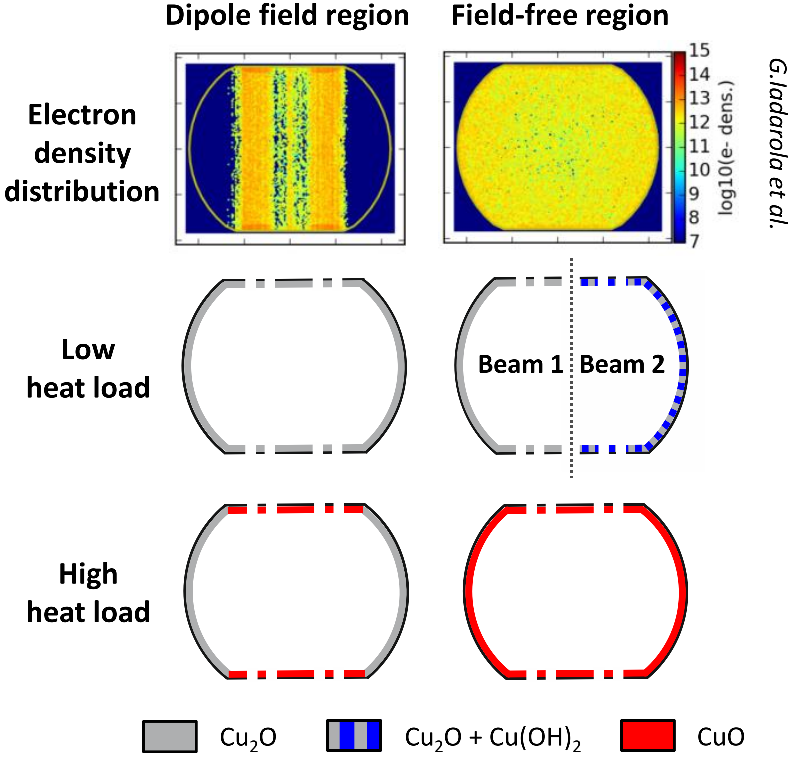
- Vanishing of $Cu(OH)_2$ (surface cleaning): confirmed even in samples extracted from LHC
- Decrease of carboxyl/carbonate contributions (surface cleaning)
- Shift of C1s peak to lower binding energy (graphitization): confirmed partially in LHC samples

Surface analysis of LHC components: extracted beam screens 1

Investigation to understand differences of heat load on cryogenics arcs of LHC related to e-cloud
May-August 2019: extraction of beam screens hosted in one high and one low heat load dipole magnet and characterisation of their surface in the laboratory
→ Surface chemistry (X-ray photoelectron spectroscopy)
→ Secondary Electron Yield
→ Electron conditioning behaviour







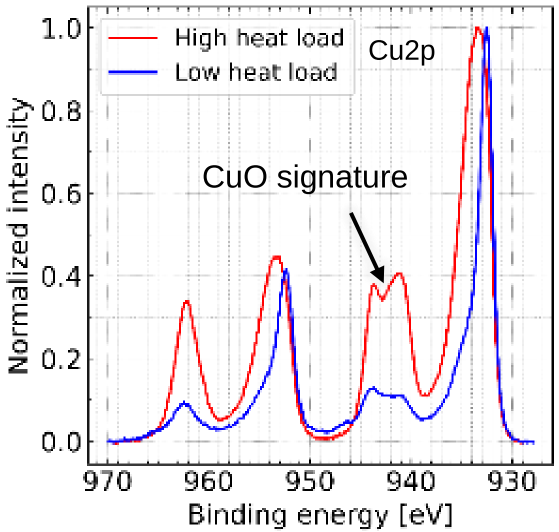
In high heat load beam screens

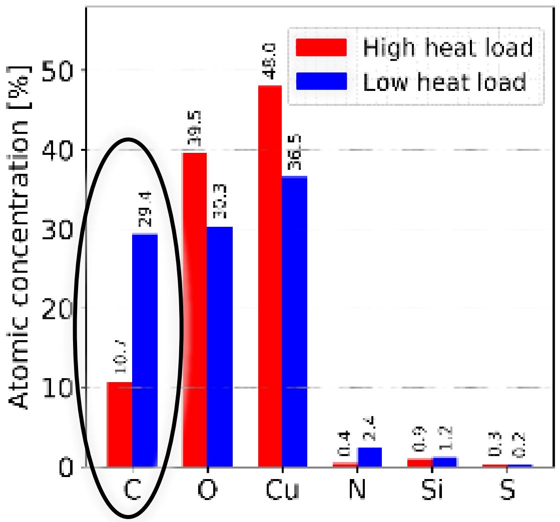
- Presence of **CuO (not native copper oxide)** with a field-related azimuthal distribution
- **Very low amount of carbon** at all azimuths

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

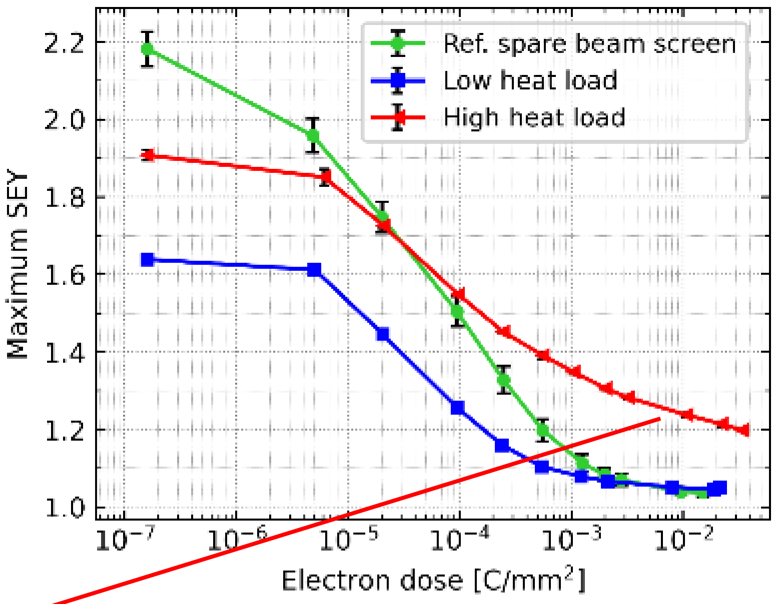
Surface analysis of LHC components: extracted beam screens 2

Differences in surface composition of beam screens extracted in LS2 from **high and low heat-load** dipoles and SEY conditioning behariour in the lab

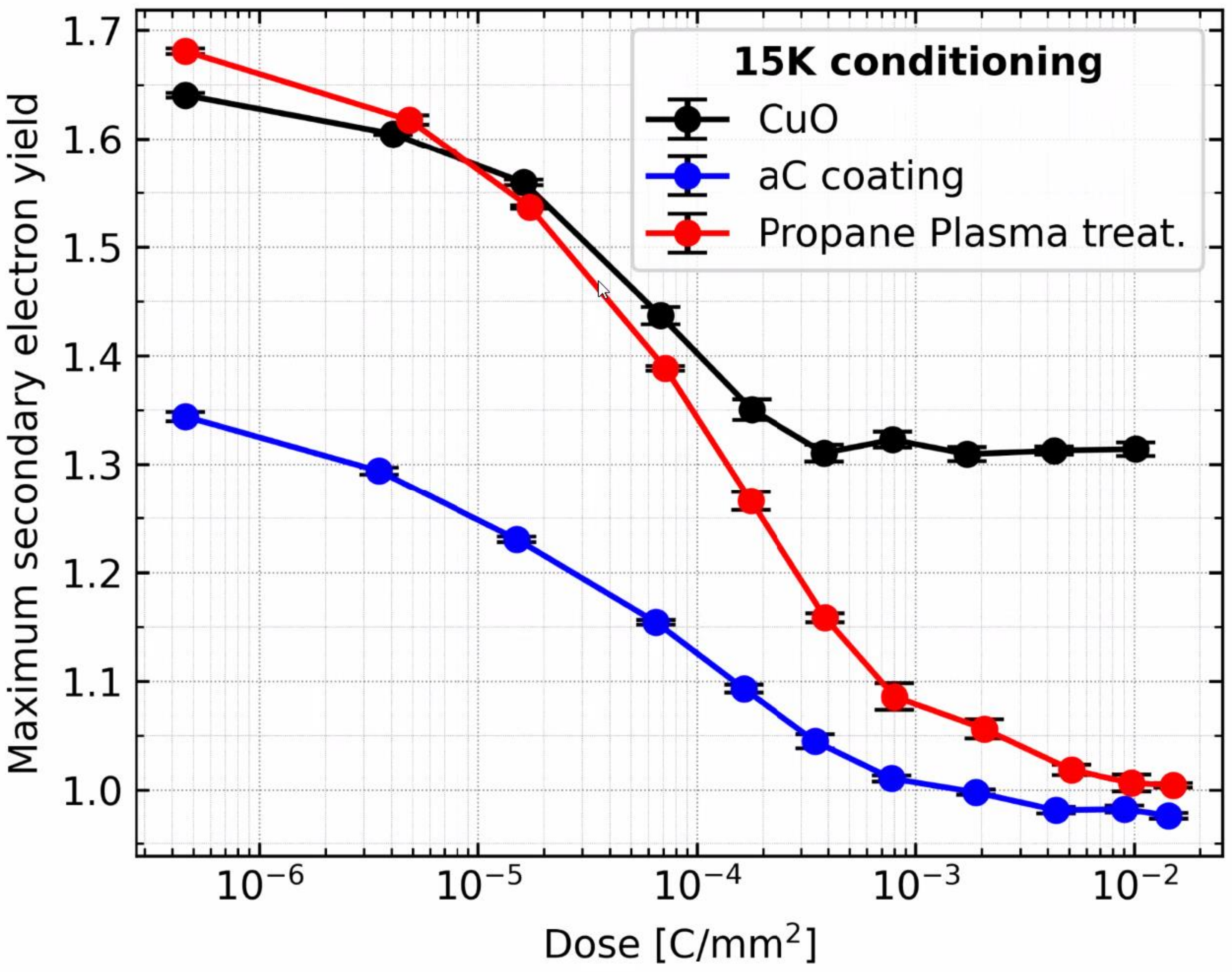




different decrease if Secondary Electron Yield with dose (=operation)



More electron cloud
More heat load


15K conditioning
CuO
aC coating
Propane Plasma treat.
Maximum secondary electron yield
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
10^{-6}
10^{-5}
10^{-4}
10^{-3}
10^{-2}
Dose [C/mm^2]