

Workshop R&Ds - Développements Instrumentaux / Virgo-ET

Chemical contamination analysis on mirror surfaces and cleaning process



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Main issues concerning test mass mirrors of ET

- **Chemical contamination of mirror surfaces** : decrease in optical performance
- **Cryosorption on cold optics (frost)** : Gas can be cryosorbed on the mirror surface inducing detrimental effects on the optics → but warming up should be avoided (time consuming)
- **Electrostatic charge of mirrors** : electrostatic charging that may induce unwanted noise : for VIRGO and LIGO optics, procedures are undertaken for neutralization with positive/neg ions → but not applicable at Low Temperature

These issues also represent interesting topics in the field of materials for accelerators (similar centre of interest)



Cryogenic Mirrors and surface quality

IJCLab contribution to investigate strategies to mitigate these detrimental effects

Mirror contamination analysis :

- X-Ray Photoelectron Spectroscopy @ 10 K
(Vide&Surfaces platform)

- MeV TOF-SIMS (collab. ANDROMEDE facility @IJCLab)



EVE
TOF-SIMS



Sample

12MeV – Au₄₀₀⁴⁺ nanoparticle beam

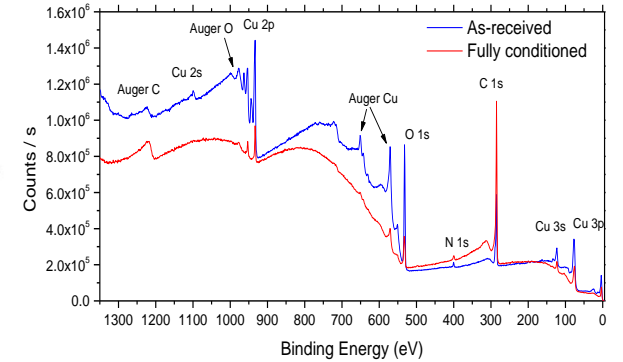


Routine XPS

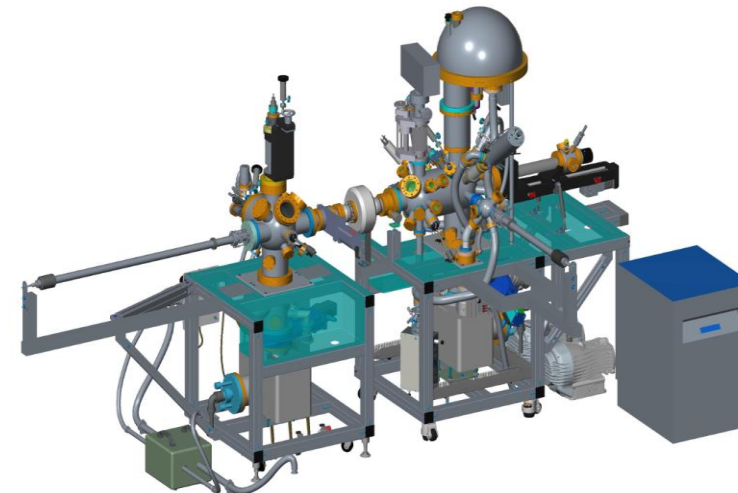


Funding : EQUIPEX+ PACIFICS

Particle Accelerators Initiative for Future Innovative and Challenging Systems



Multi technical set-up – Surface analysis at cryogenic temperature



SEY
XPS
RGA
ion gun
@ 10K

2024





Main issues concerning test mass mirrors of ET

- **Chemical contamination of mirror surfaces** : Andromede analysis performed by Isabelle Ribaud
 - **Cryosorption on cold optics (frost)** : Gas can be cryosorbed
 - **Electrostatic charge of mirrors** : electrostatic charging induce unwanted noise
- } 1st test on a Cluster ion gun used to neutralize and clean mirrors @ RT at Versailles University



- **Purpose of the analysis**

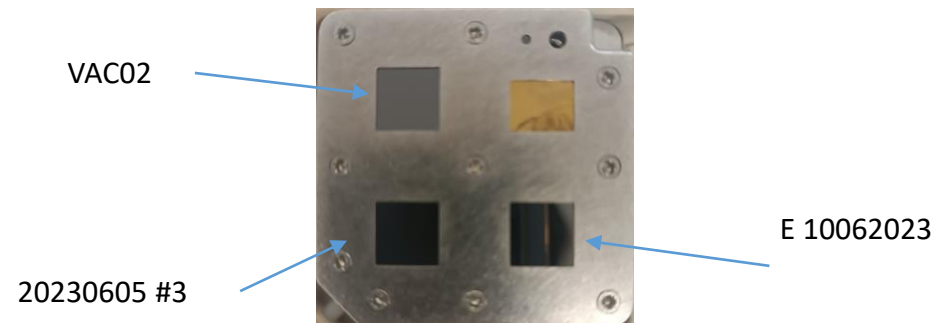
3 Si-wafers were used to collect contaminations inside a Virgo tower

the interest is to estimate the chemical composition of the particles present in greater numbers.

- **Samples description : 3 Si-wafers**

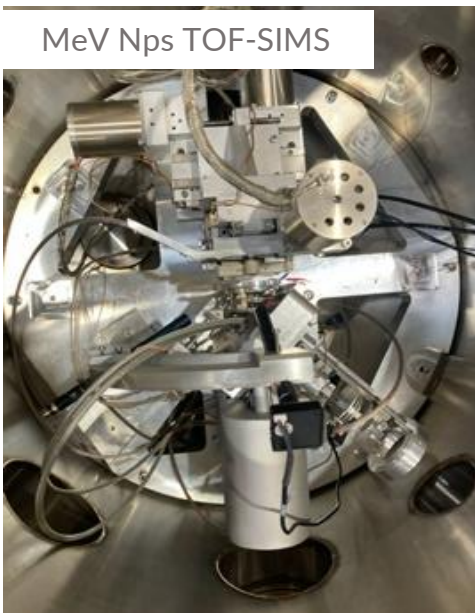
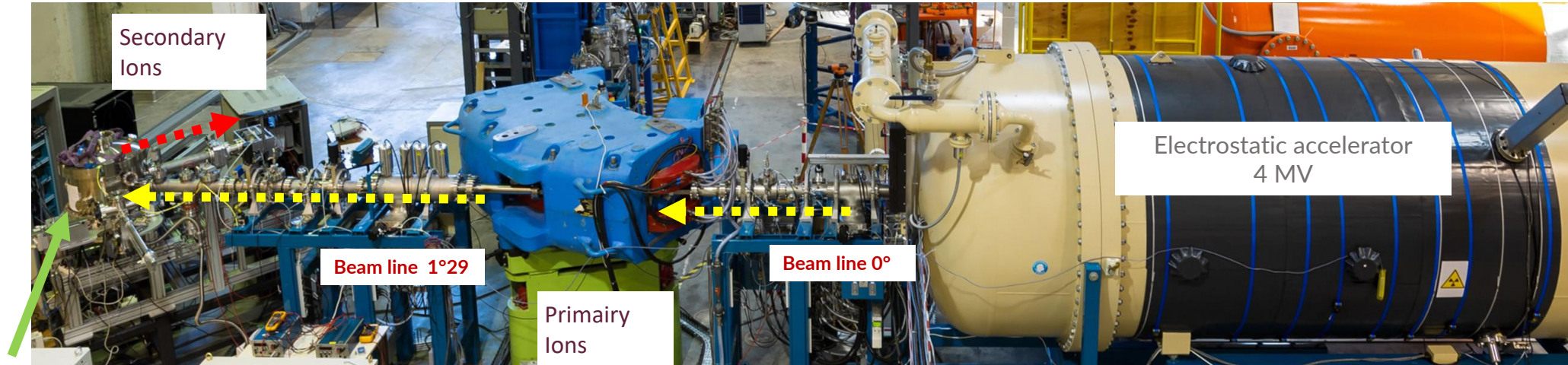
- 20230605 #3 exposed to a vacuum cycle , order of 20 particles > 10 um
- E 10062023 - exposed to operator activities, order of 100 particles > 10 um (central surface 5 x 5 cm²)
- VAC02, CO₂- exposed to particles fallout during cleaning - order of 300 particles > 10 um

The wafers have been cut to 10 x 10 mm to fit into the sample holder (MAVERICS)





First results on Virgo samples



mosaic

Probe

12 MeV Au₄₀₀⁴⁺ nanoparticles

Measurement conditions

Ø 200µm

Andromède

ToF EVE Mass Spectrometer

IJCLab - FIIRST

Data Acquisition

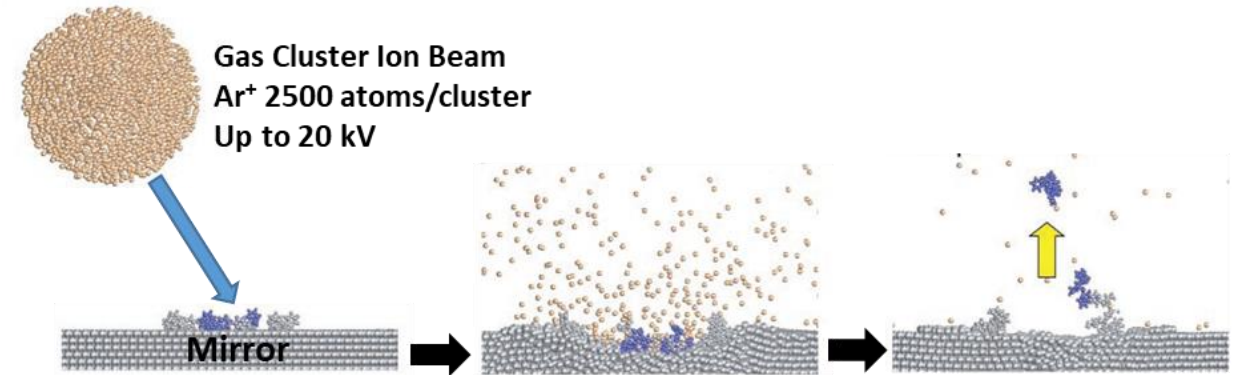
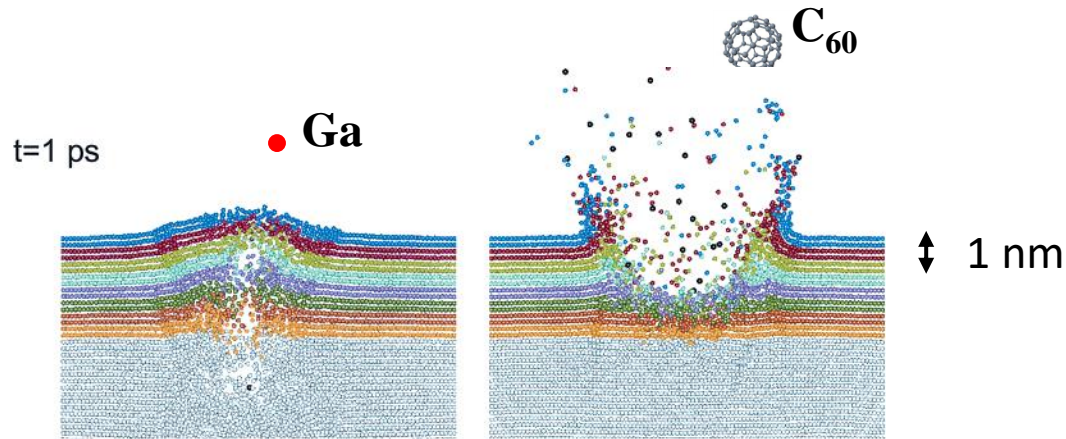
Decod-Narval

Data Analysis

C-Visu

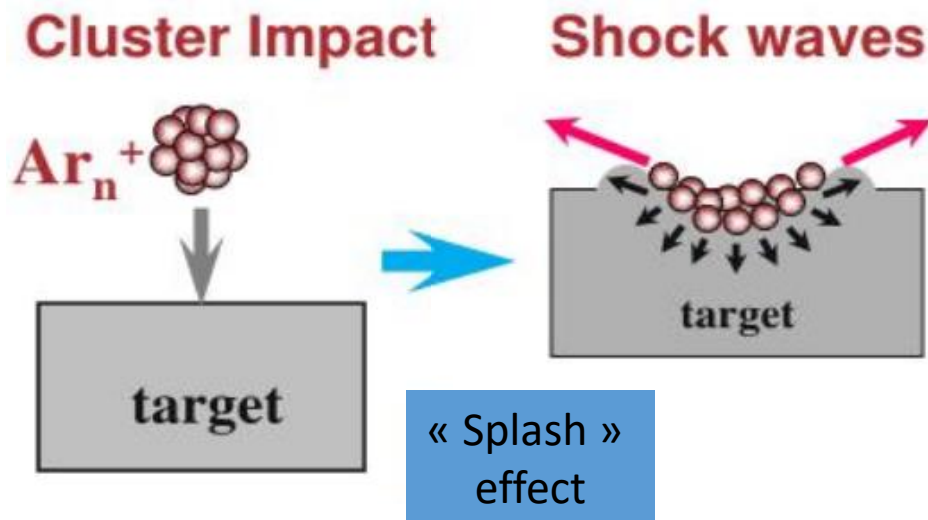


Using the Cluster ion beam cleaning : mirrors cleaning/ice removing?



Postawa Z. et al. *J. Phys. Chem. B* **2004**, 108,7831-7838

Garrison B. J. & Postawa Z. *Mass Spectrom. Rev* **2008**, 27, 289-315

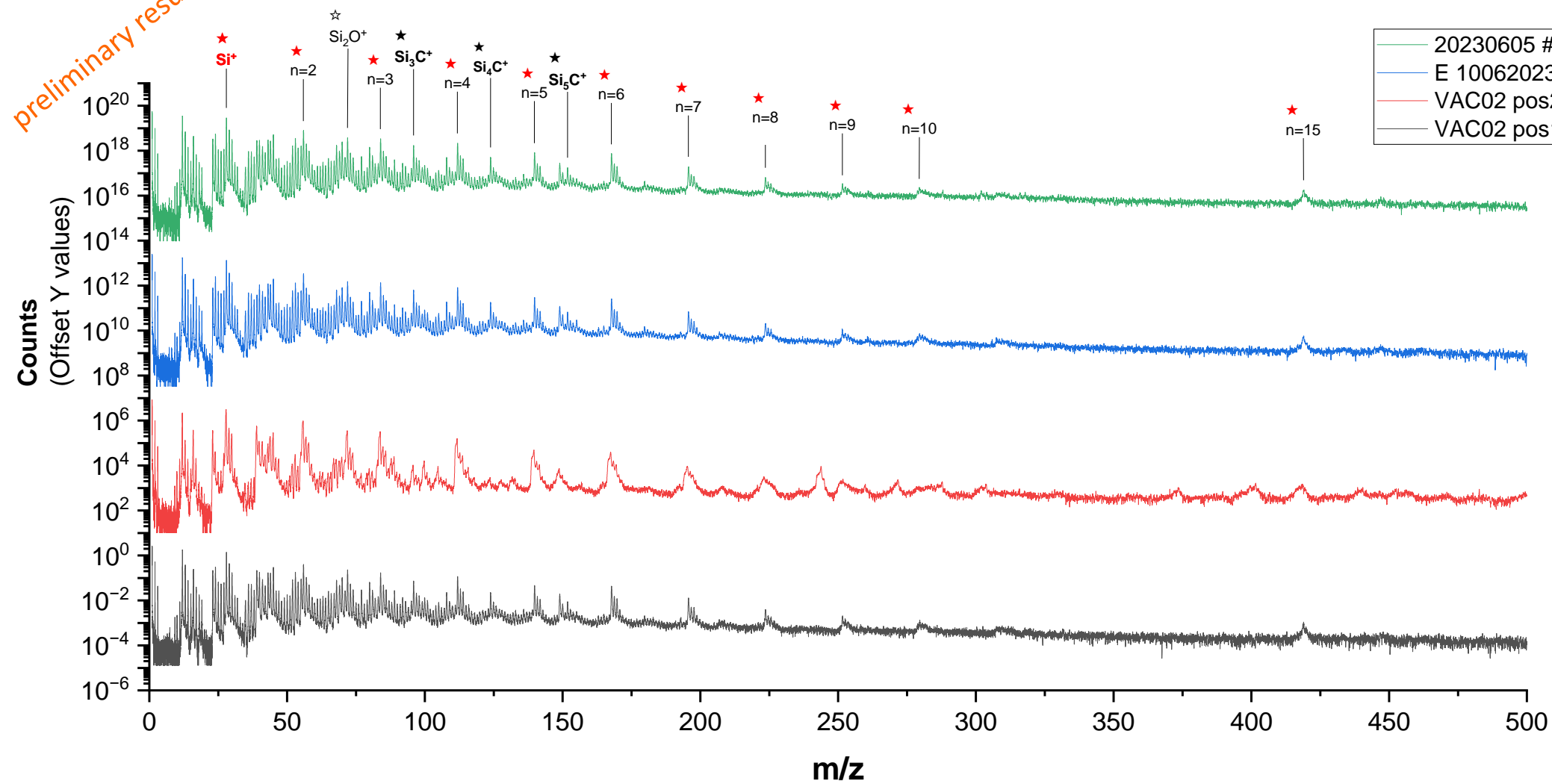


- Gas Cluster Ion Beams (GCI) impact a surface with very low energy, down to as little as 1 eV per atom.
- At such low energies they sputter material without modifying the surface chemistry, i.e. without breaking bonds
- Cleaning effect without materials damage (unlike single ions)
- Sputtering of organic layers without destroying the underlying layers (only the extreme surface is impacted)



Positive secondary ion mass spectrum (NPs MEV TOF- SIMS)

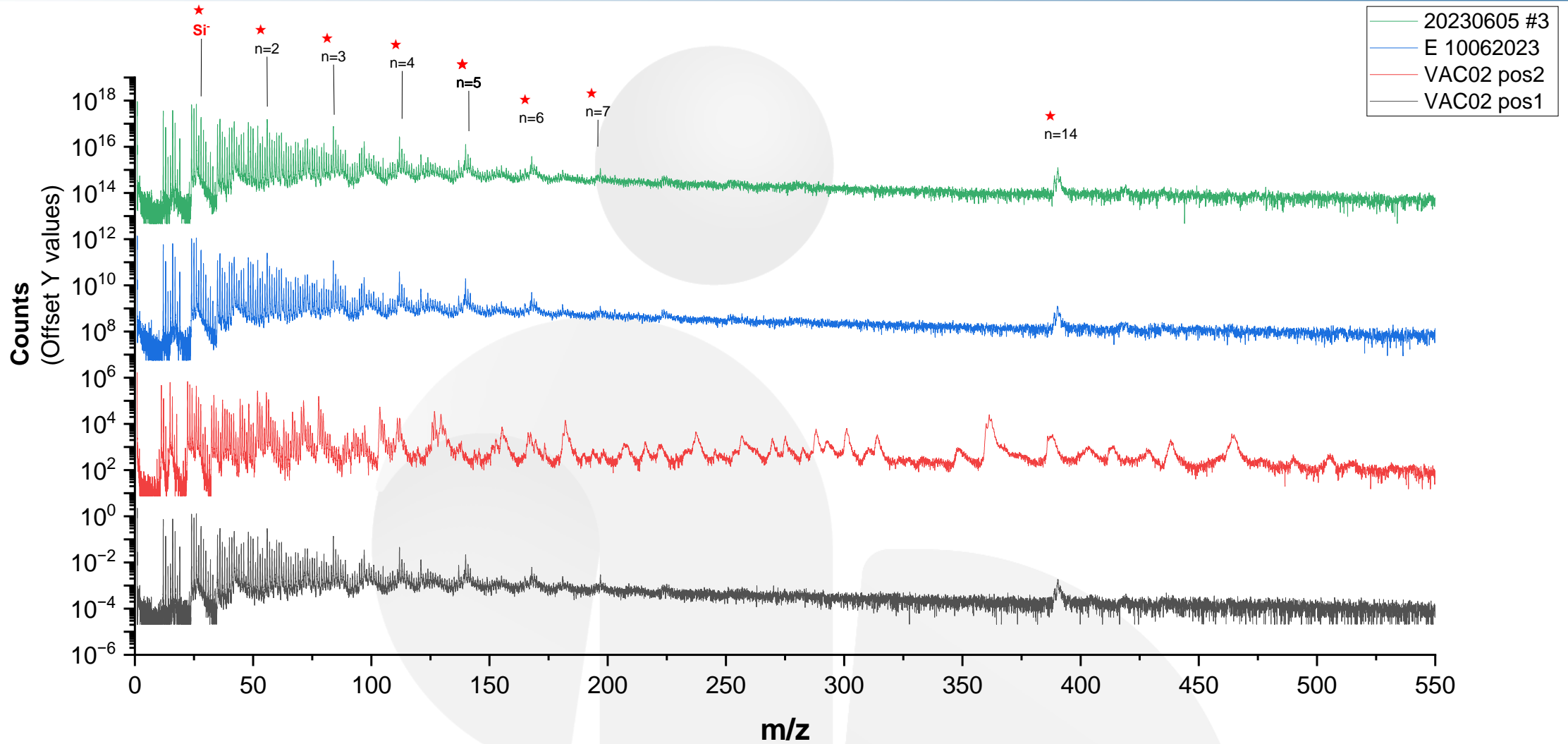
preliminary results



Isabelle Ribaud



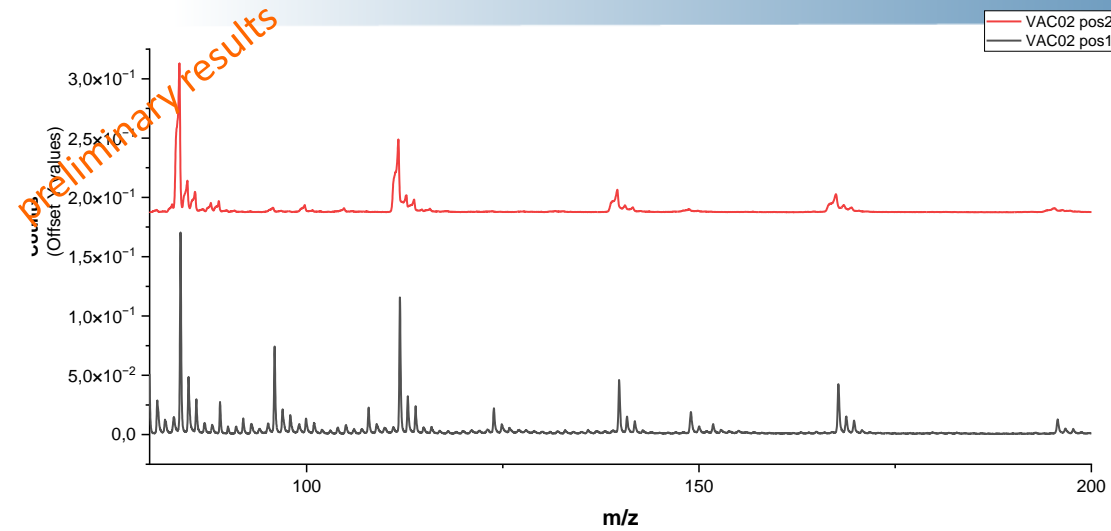
Negative secondary ion mass spectrum (NPs MEV TOF- SIMS)



→ Spectral analyzes show surface pollution coverage (200 μ m) on the sample VACO2 Pos 2



Zoom of the positive mass spectrum

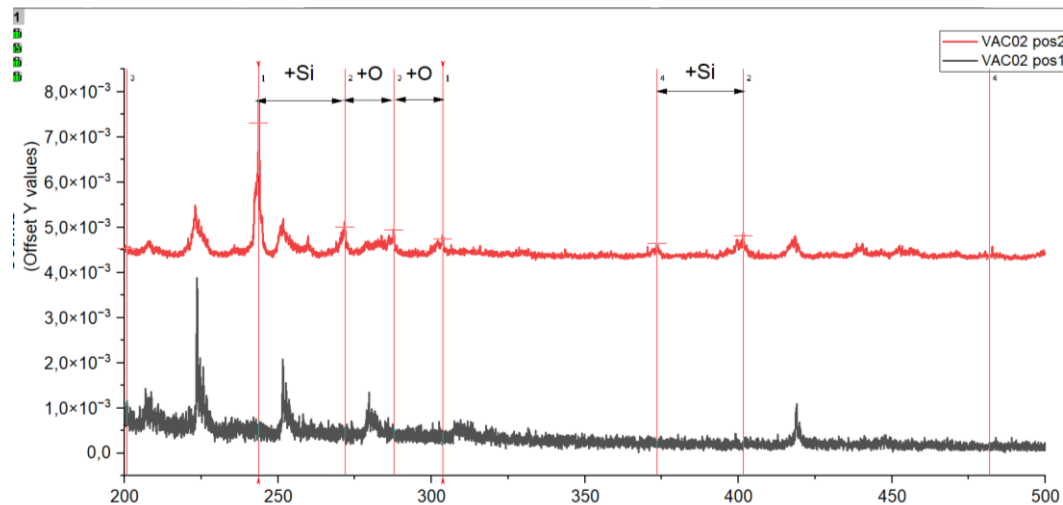


Sample :

VACO2, CO₂- Order of 300 particles > 10 μm

VACO2 Pos2/Pos1

Decrease of the Si_nC⁺ Clusters



VACO2 Pos2/Pos1

New peaks with
Si, O and O₂ adducts

Presence of a Particle or partial coverage? or local
modification of the surface?

Isabelle Ribaud



Confocal microscopy 3D imaging

Objective lens x50

Few images of scan
(Laser + Optical)

Close-up of particles or
surface defaults. All 3D
profiles have a z axis factor
magnified for clarity

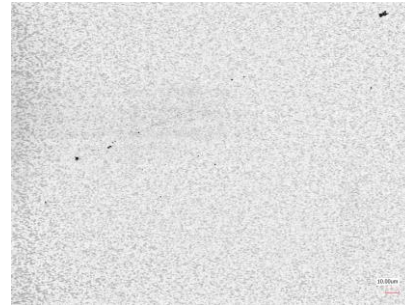
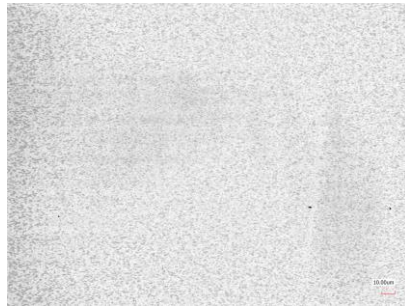
Various μm defects at the
surface

*Difference of the particle
density ?*

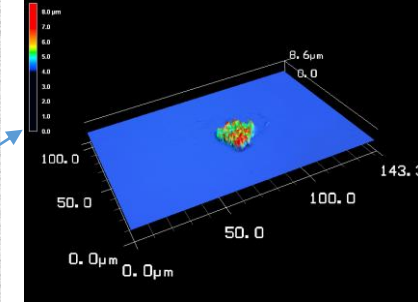
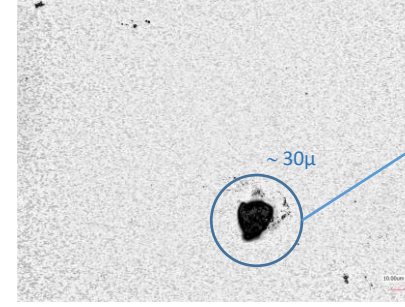
Images were acquired
after TOF SIMS analysis...

→ *Checking original
samples*

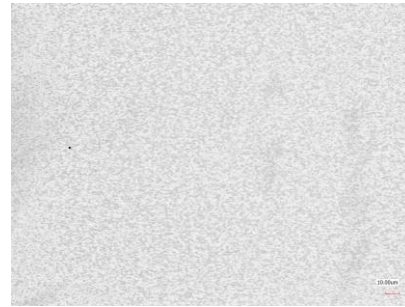
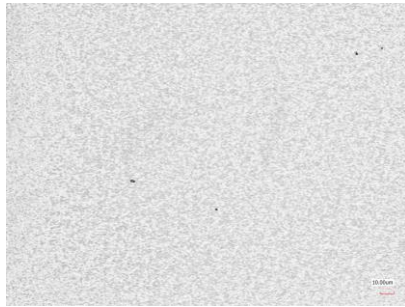
- 20230605 #3 order of 20 particles > 10 μm



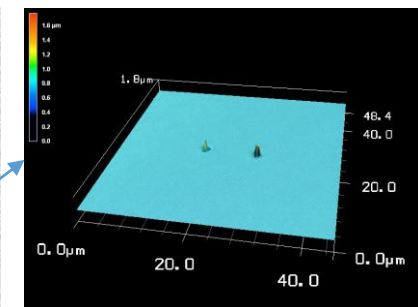
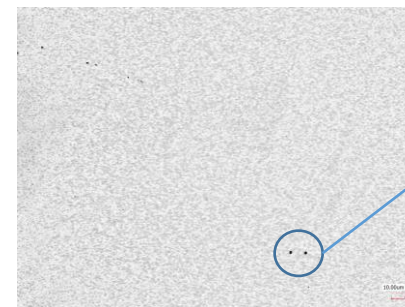
- 1 particle > 10 μm were observed over an area of 2 mm^2 (scan).



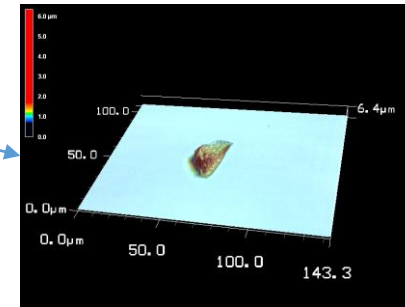
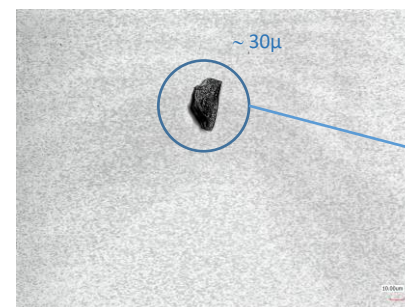
- E 10062023 order of 100 particles > 10 μm



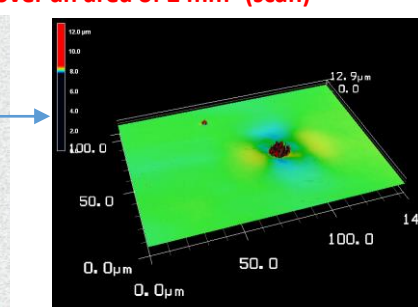
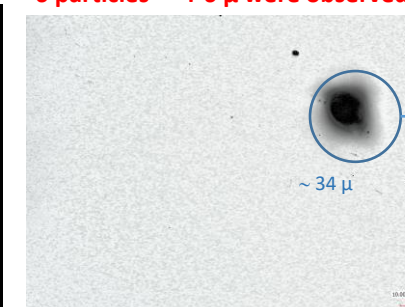
- 0 particles > 10 μm were observed over an area of 2 mm^2 (scan).



- VAC02, CO2 - order of 300 particles / > 10 μm



- 2 particles > 10 μm were observed over an area of 2 mm^2 (scan)
6 particles ~ 4-6 μm were observed over an area of 2 mm^2 (scan)





- New analysis need scan of the surface on a large area to 2mmx 2mm or more ?
- Is it possible to provided Si wafer without exposure to particles to compare?
- Smaller samples are required to avoid a cutting process when analyzing particulate contamination.



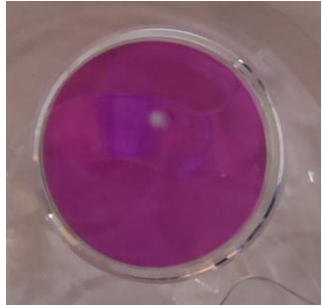
- New surface treatments in ultra high vacuum by **an ion cluster gun**:
 - **To neutralize unwanted electrostatic charge on test mass mirrors**
 - **To remove cryosorbed gas**
 - **To clean the mirror surface**

without modifying or damaging the solid surface (no deterioration in mirror performance is expected)

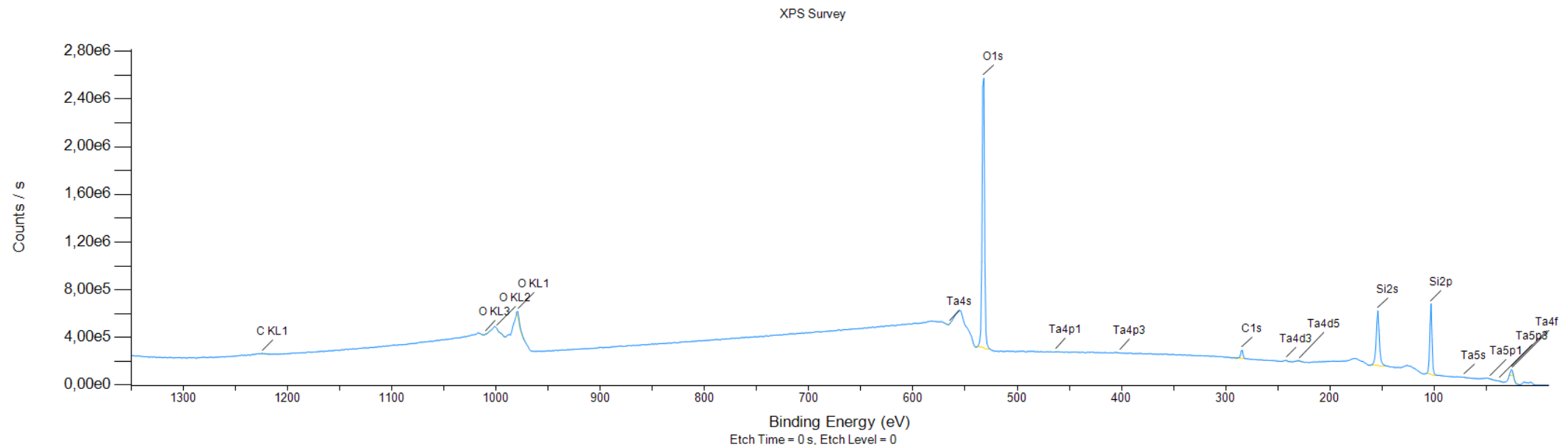
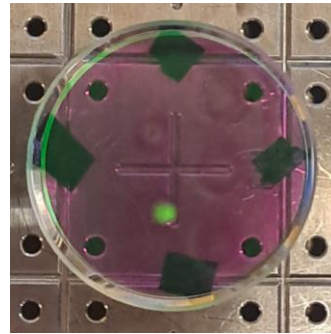
1st test using an ion cluster gun @ RT (Versailles University) to check if the cleaning deteriorate mirror performances



X-ray photoelectron spectroscopy



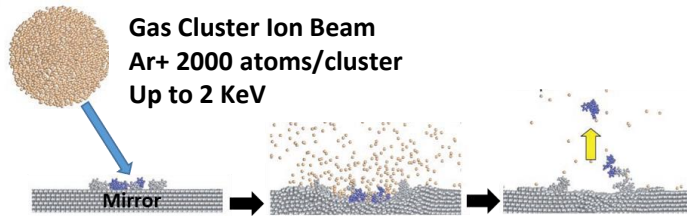
Mirror
1 inch



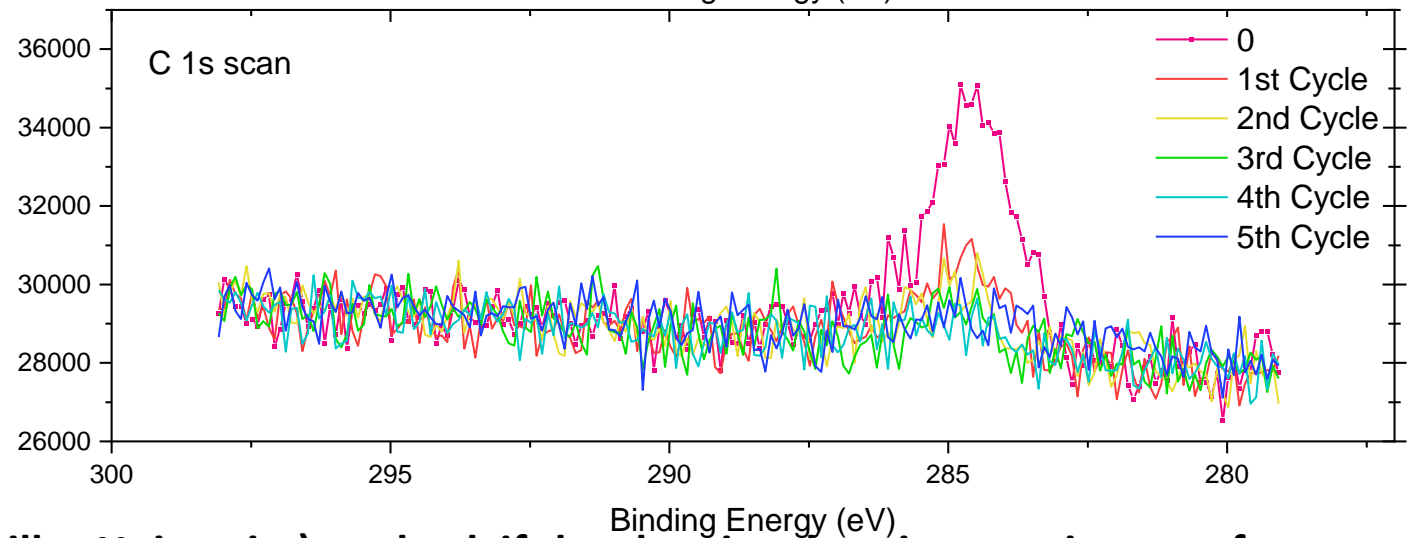
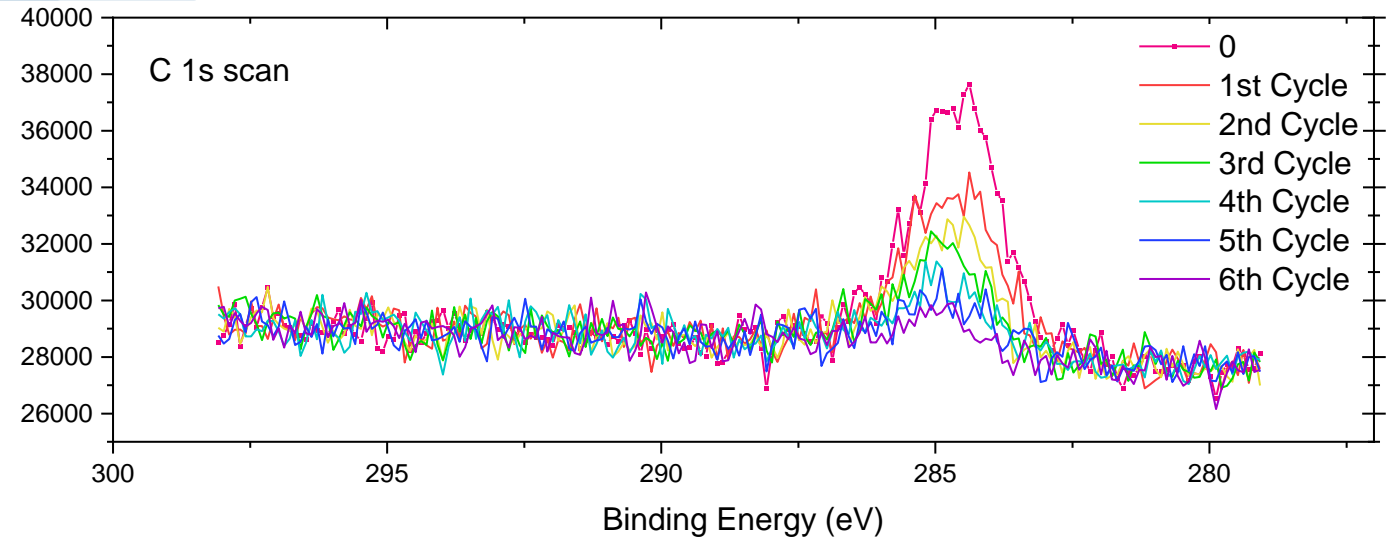
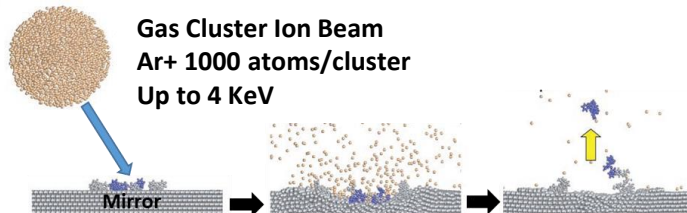


Cleaning process using an ion cluster gun

1st parameters



2nd parameters



1st test using an ion cluster gun @ RT (Versailles University) to check if the cleaning deteriorate mirror performances

→ The optical properties were checked after cleaning and the optical properties remained unchanged



➤ Chemical contamination of mirror surfaces :

- New analysis need scan of the surface on a large area to 2mmx 2mm or more ?
- Is it possible to provided Si wafer without exposure to particles to compare?
- Smaller samples are required to avoid a cutting process when analyzing particulate contamination.

➤ New surface treatments in ultra high vacuum by **an ion cluster gun**:

- **To neutralize unwanted electrostatic charge on test mass mirrors**
- **To remove cryosorbed gas**
- **To clean the mirror surface**

1st cleaning test using an ion cluster gun @ RT (Versailles University) → Mirrors optical properties OK

Best parameters for the cleaning process has to be find (Cluster size, energy, step, etc.) using new samples



- Chemical contamination of mirror surfaces :
 - A specific sample holder must be developed to analyze 2 inch mirrors on Andromede Facility
- New surface treatments in ultra high vacuum by **an ion cluster gun**:
 - A new cleaning process will be carried out on the polluted mirror to recover good optical properties

Thank you for your attention