UHV characterization of advanced materials and their coatings

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1st Workshop of ARIES PowerMat (WP17)

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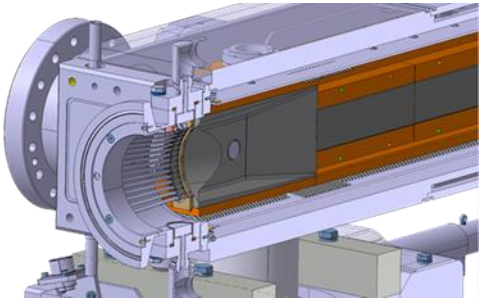
CERN | EN ENGINEERING DEPARTMENT ARIES

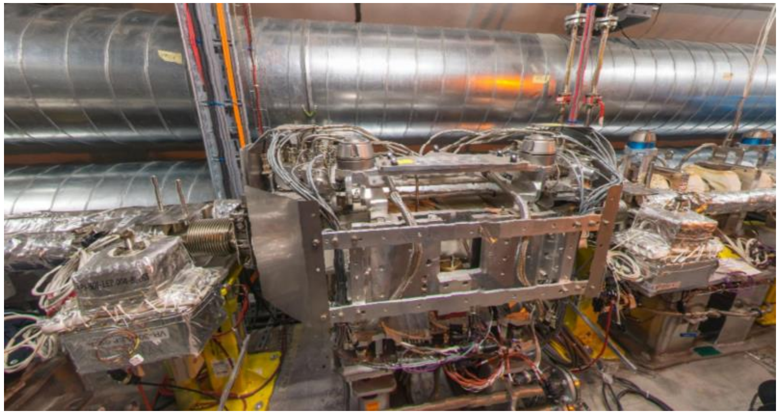
Outline

- Introduction: the LHC and the UHV
- Outgassing of collimator materials:
  - Graphite, CFC and MoGr
  - MoGr investigation – bulk material
  - MoGr investigation – coating
  - CuCD
- Conclusions and outlook

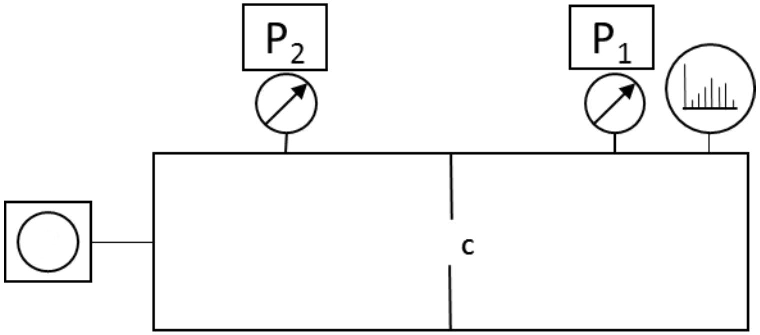
The UHV requirements

- In all the particle accelerators an high or ultra-high vacuum is required: beam stability and lifetime (beam-gas scattering)
- LHC vacuum acceptance limits for collimators:
  - Low outgassing rate ($1 \cdot 10^{-7}$ mbar·l/s)
  - No NEG saturation (N_2, CO, CO_2,..)
  - Low SEY (ESD, electron cloud)





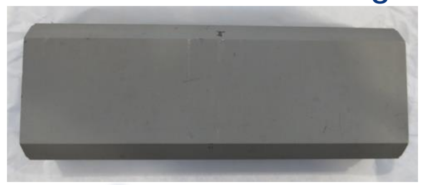
$$Q = (P_1 - P_2) \cdot C$$



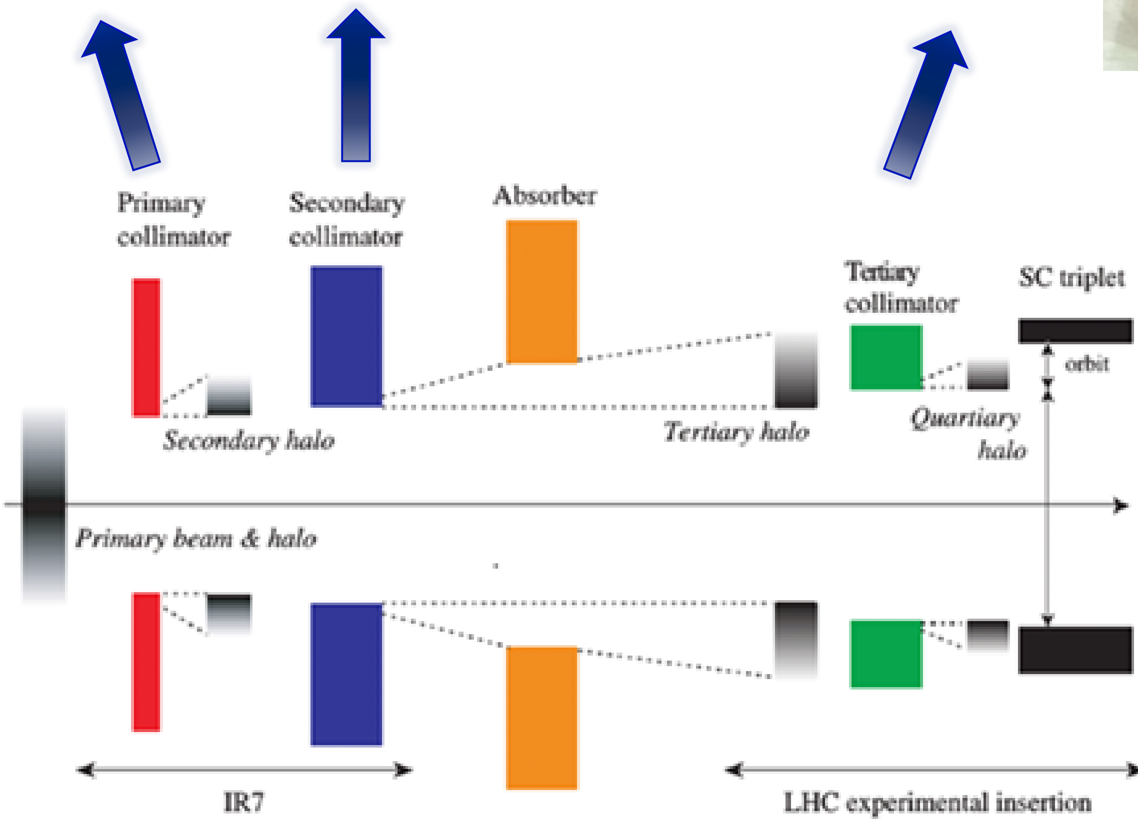
Collimation system upgrade

MoGr

MoGr with Mo coating

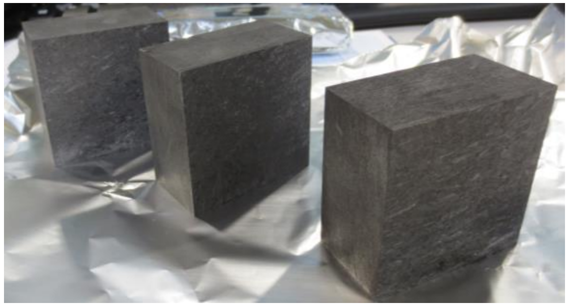


CuCD

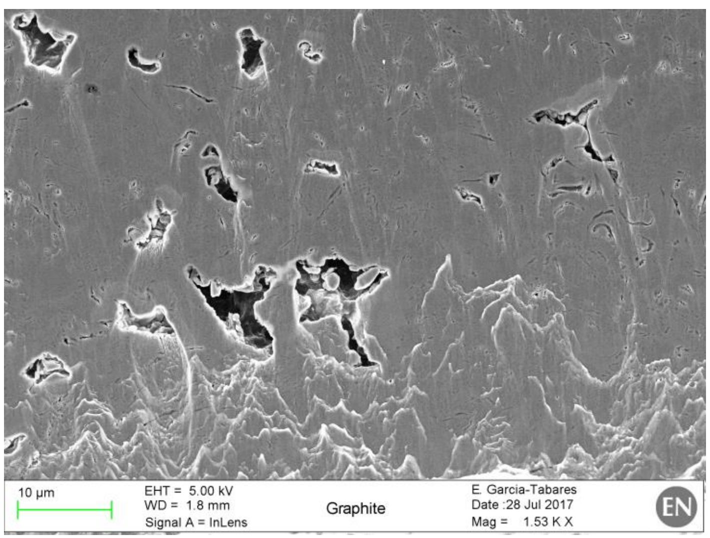


Graphite outgassing – Air content

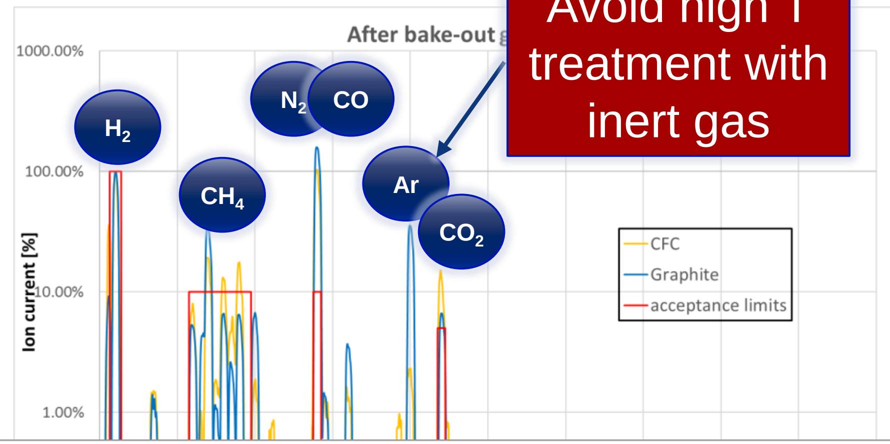
2D Carbon-Fibre-reinforced Carbon (CFC)

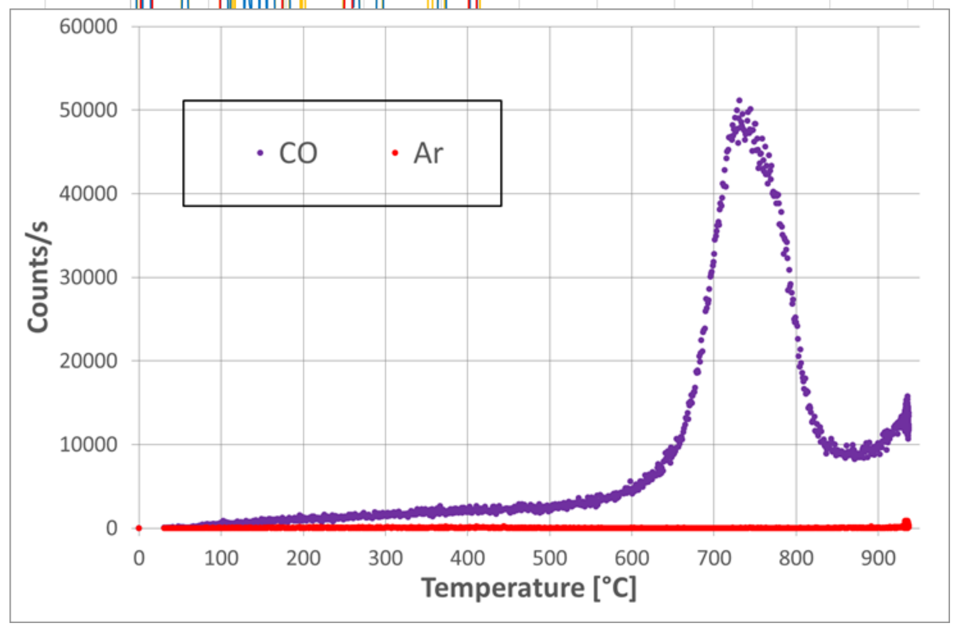


Graphite



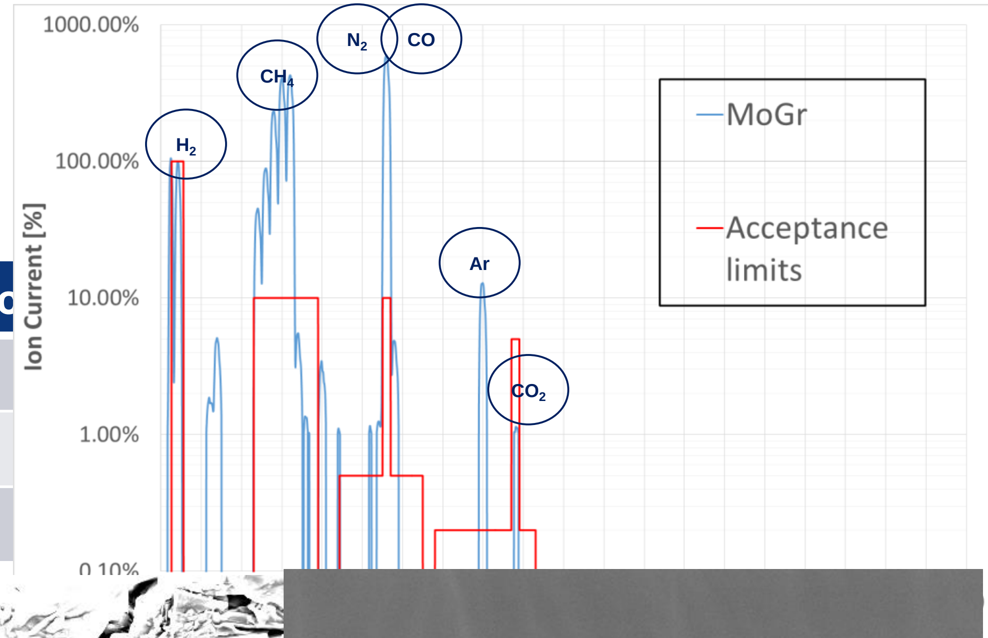
Avoid high T treatment with inert gas

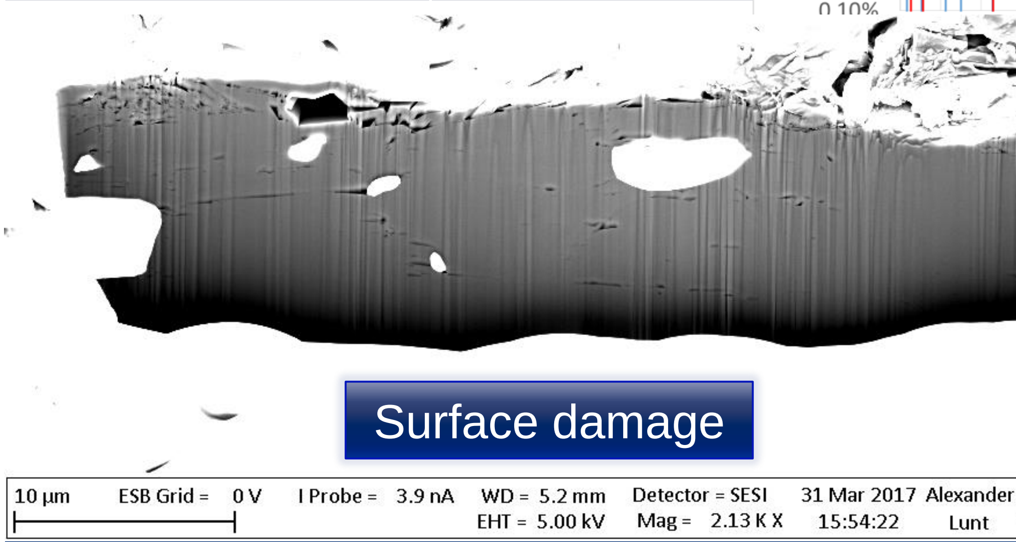


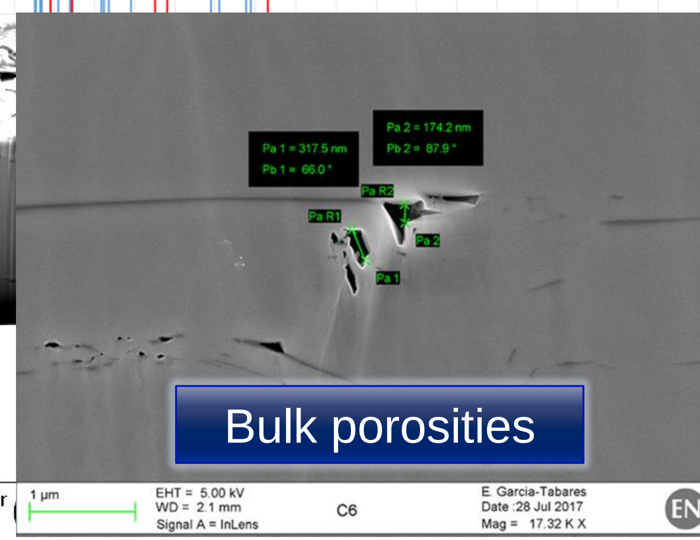


Molybdenum-Carbide Graphite

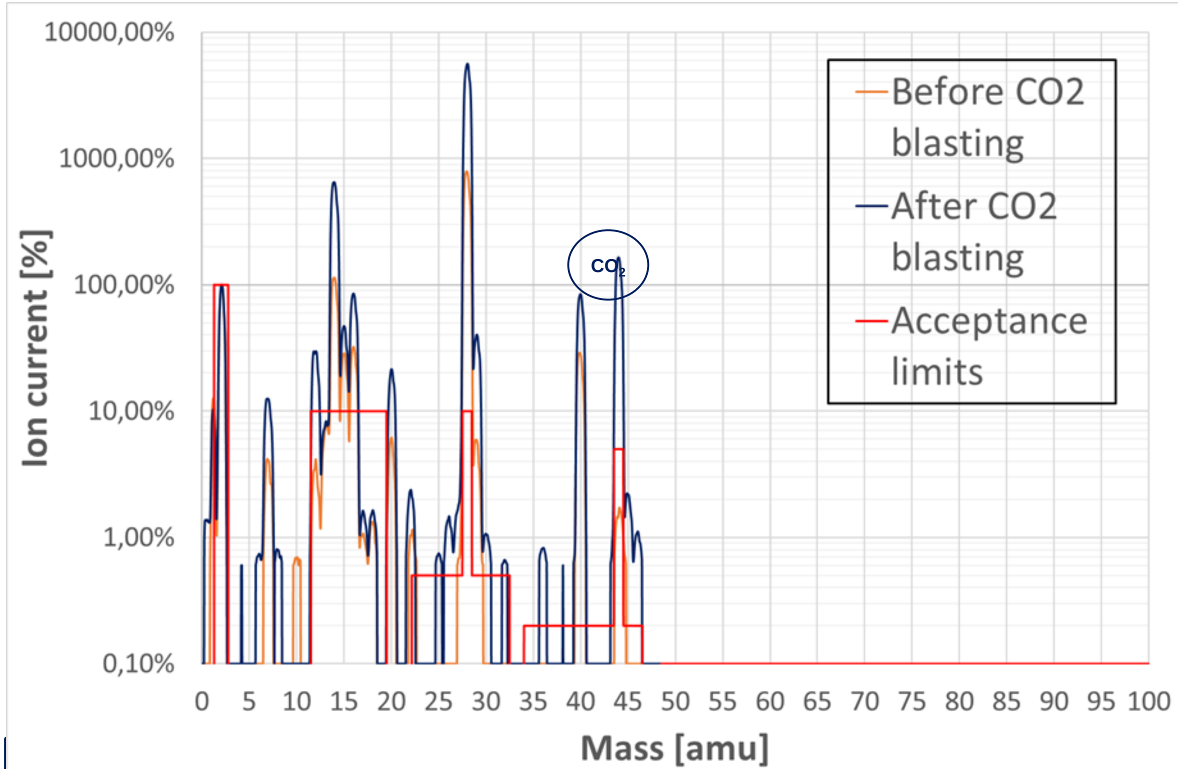
Material	Q_s [mbar·l/ s·c
MoGr	$1.9 \cdot 10^{-10}$
Graphite	$1.3 \cdot 10^{-11}$
CFC	$1.9 \cdot 10^{-11}$

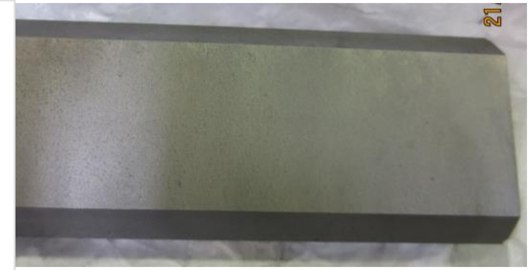


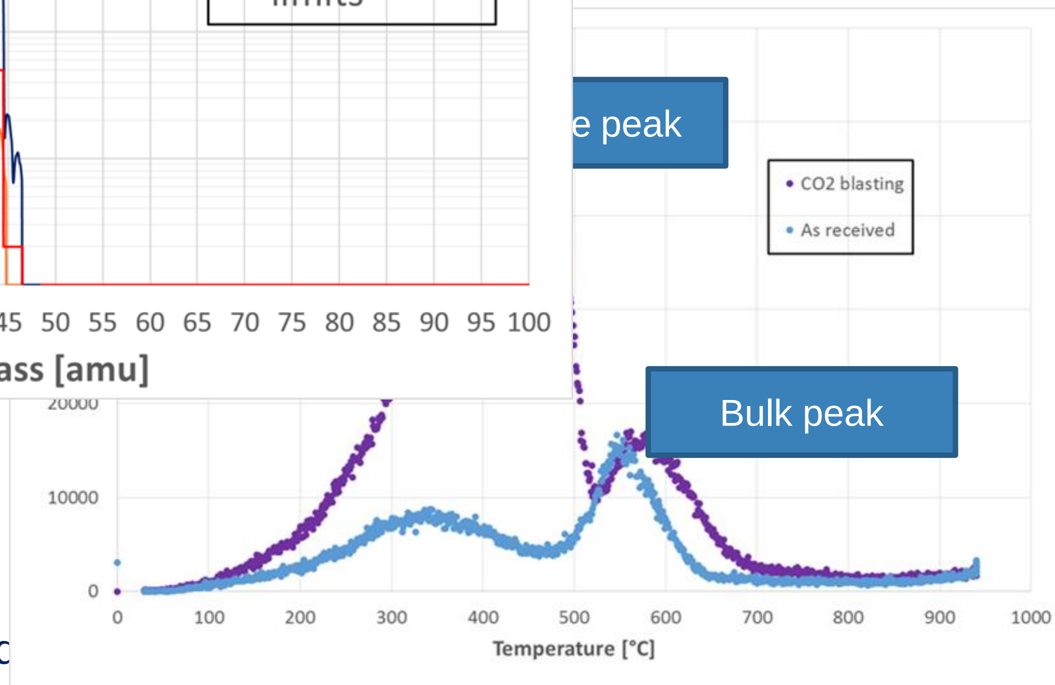




MoGr treatment



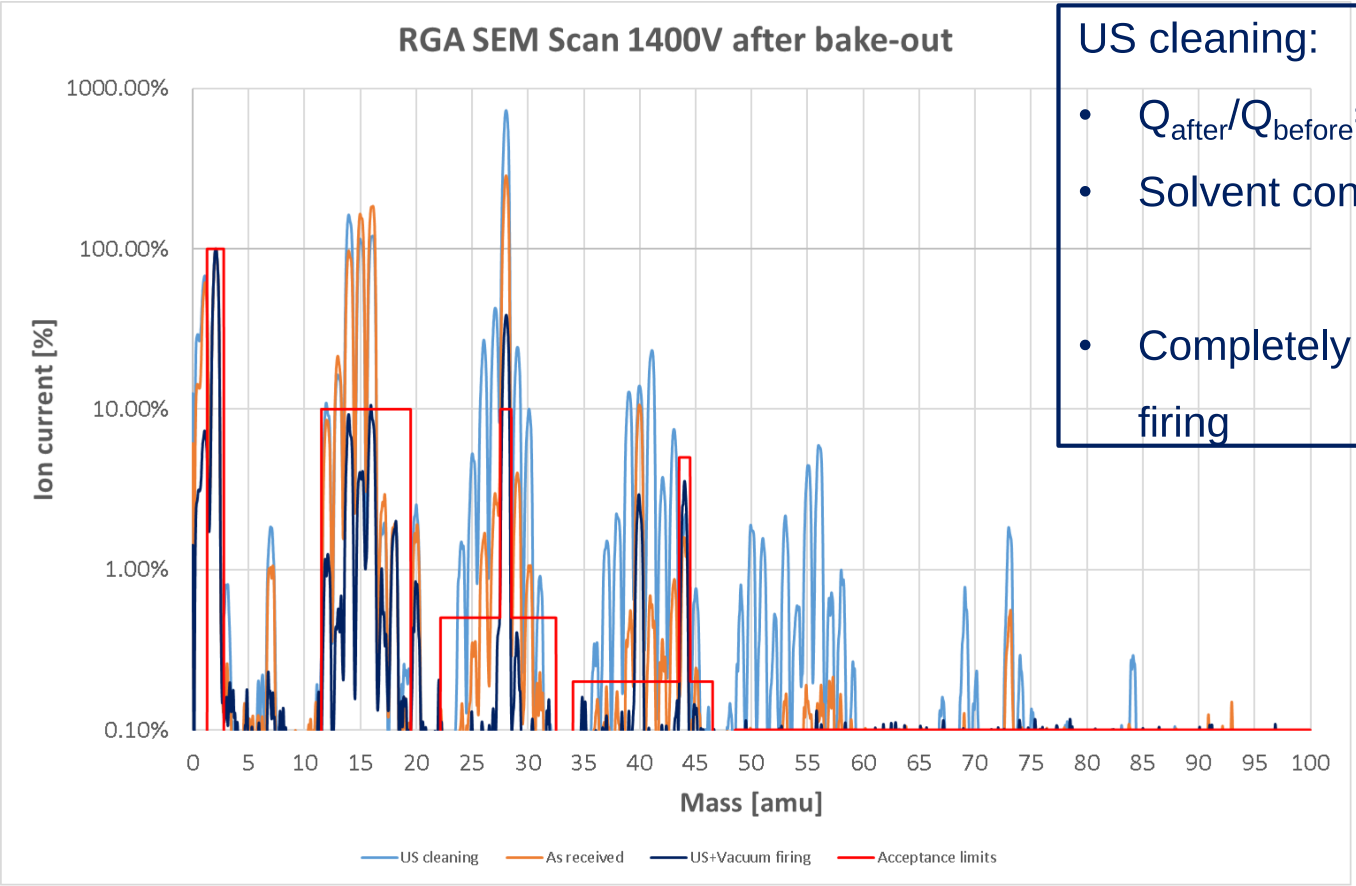




CO_2 blasting:

- Q_{after}/Q_{before}= 50
- CO_2 adsorption
- Air content (N_2, Ar) increased

US cleaning-last grade



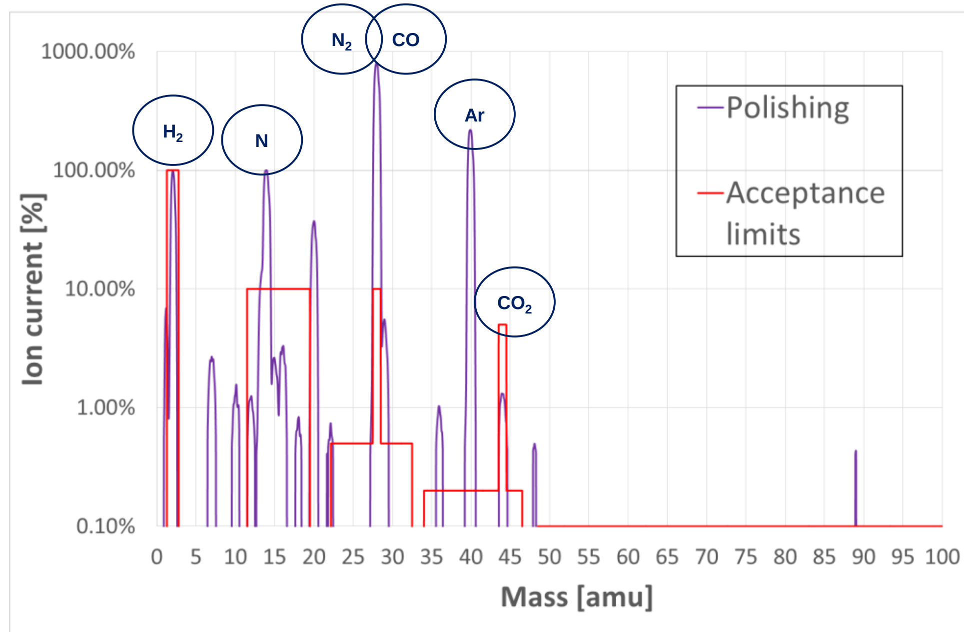
US cleaning:

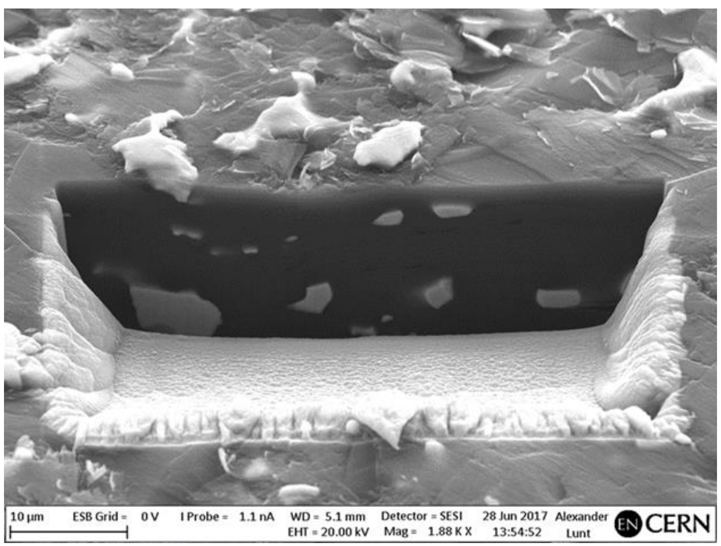
- $Q_{after}/Q_{before} = 3$
- Solvent contamination
- Completely recovered after 48h firing

MoGr treatment

Diamond paste polishing (particle size 1μm):

- Q_{jaws}= $5.4 \cdot 10^{-7}$ mbar·l/s ☒
- Air still dominant
- Surface damage seems removed



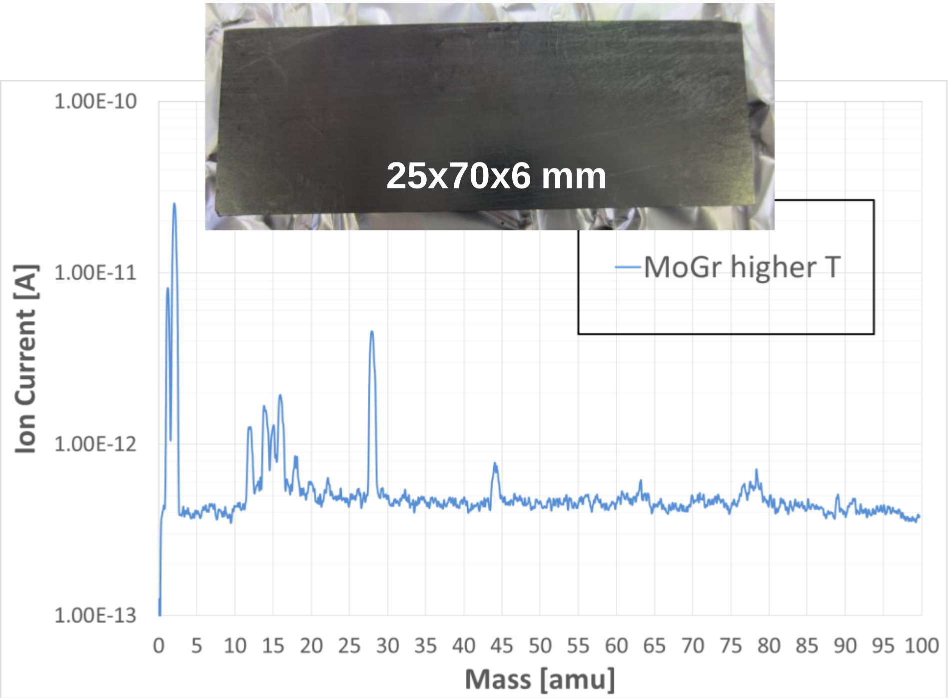


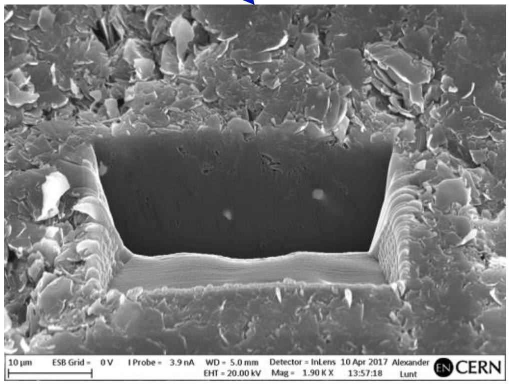
The **surface plays a role** (CO_2 blasting), but there should be also a **bulk contribution** to air trapping

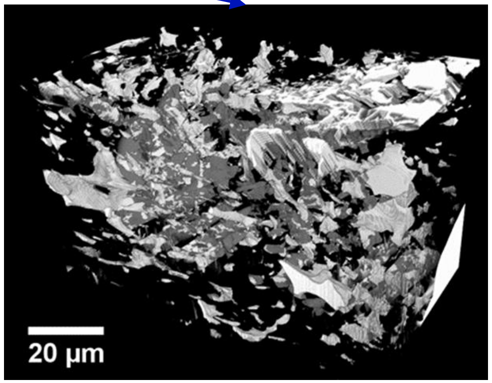
Solution 1: Higher T process

Higher temperature during production process:

- Q_{jaws}= 3.1·10^{-8} mbar·l/s ☑
- H_2 dominated
- Similar surface damaged (FIB)
- Lower density: similar void fraction, lower carbide content (3D FIB)

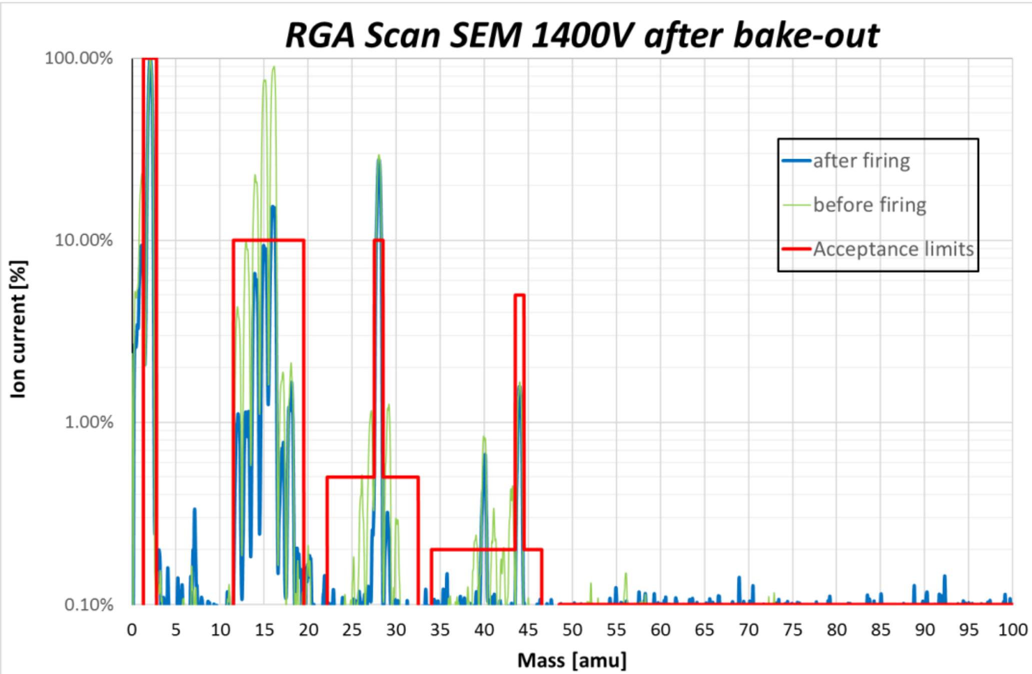


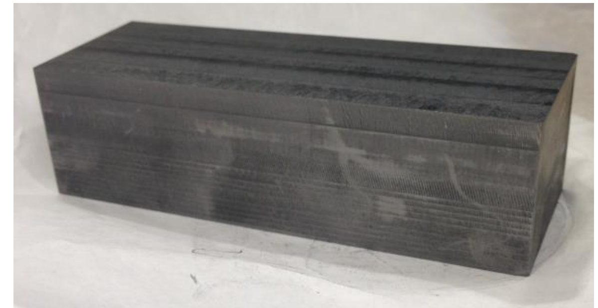




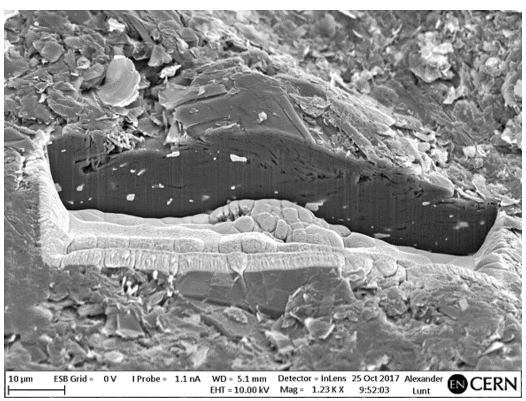
	Compliant sample	Non-compliant sample
%vol carbide	4.5	7.8
%vol void	5.1	5.7

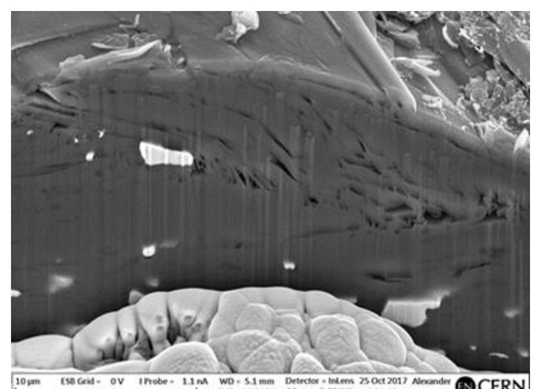
Solution 2: Different compaction



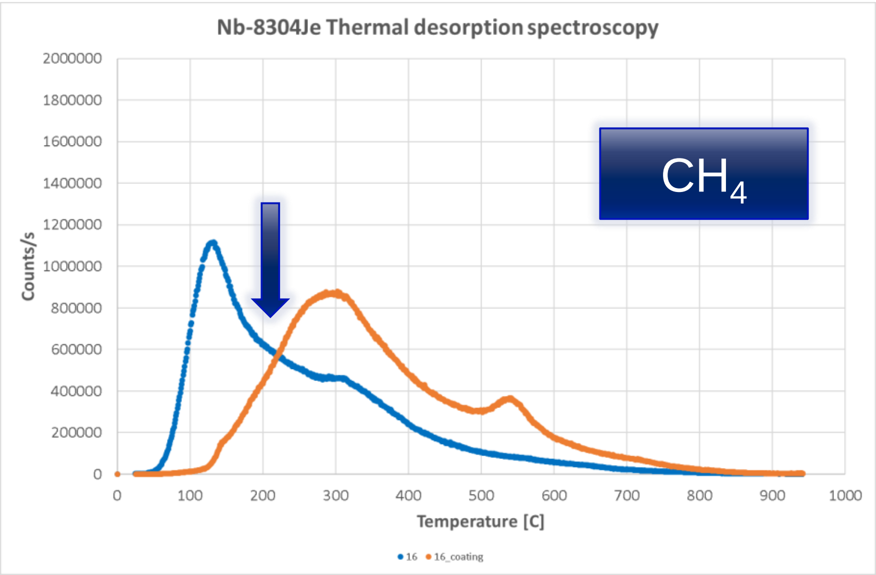


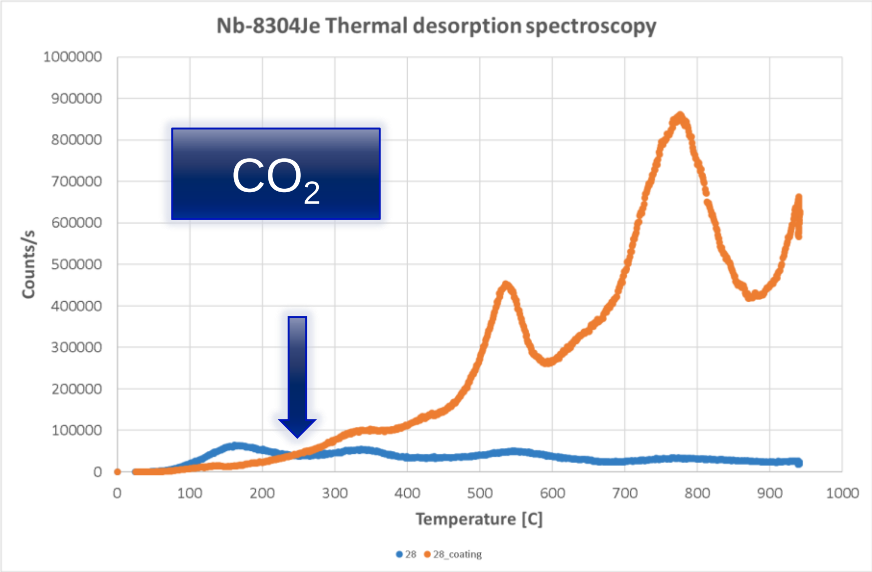
Material	Q_s [mbar·l/ s·cm^2]
As received	$4.7\cdot10^{-11}$
48h firing	$3.5\cdot10^{-12}$

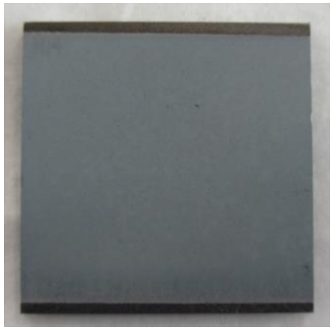




Mo coating on MoGr – TDS





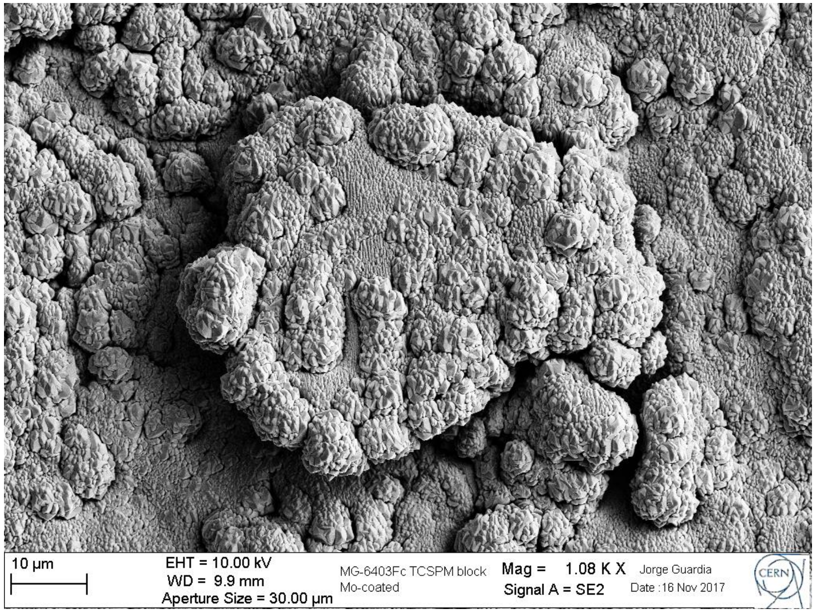


CO_2 blasted MoGr with Mo coating

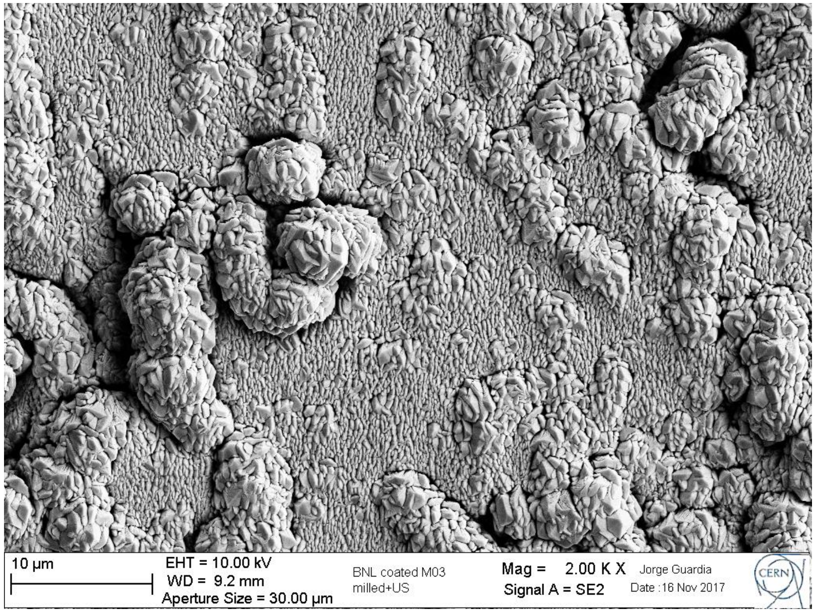
- The coating:
  - reduces the outgassing up to 200-250°C (oxide layer?)
  - increases the outgassing above these T (CO_2 blasting) ➡ with a proper preparation should be compliant

Mo coating on MoGr – SEM

- Need to verify possible air trapping within the Mo layer



CO_2 BLASTING

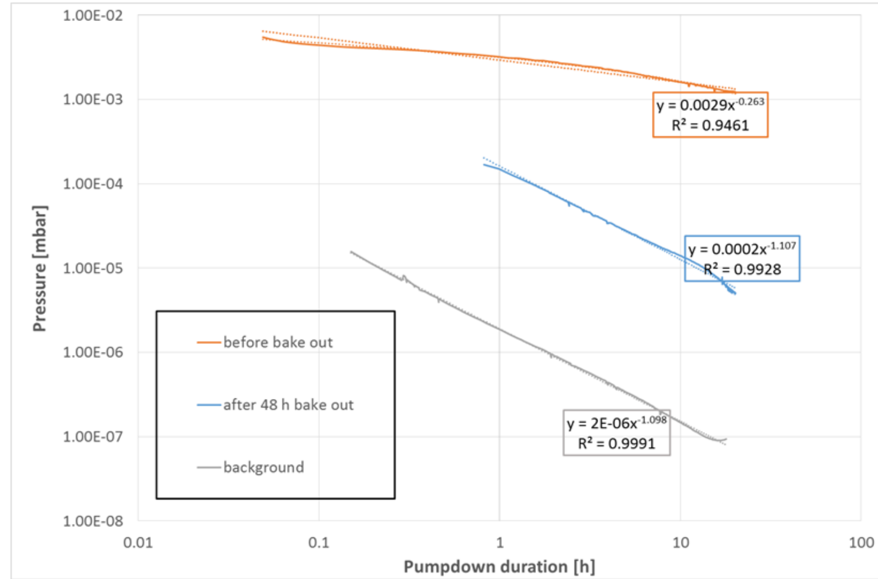


US CLEANING

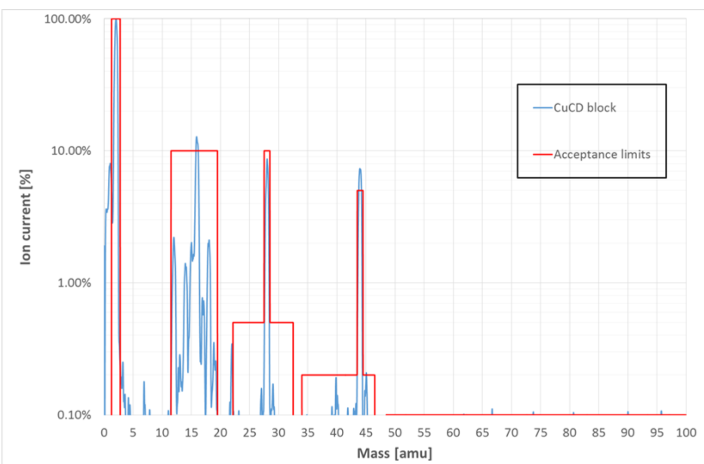
Copper-Diamond

- Robustness against accident situation: W alloy ⟶ CuCD

- US cleaning before the test→ impossible to pump-down



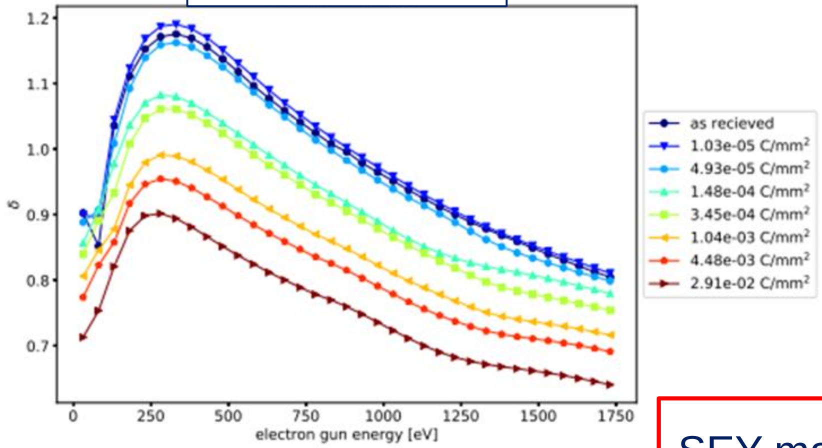
- After 48+24 h of bake-out at 250°C



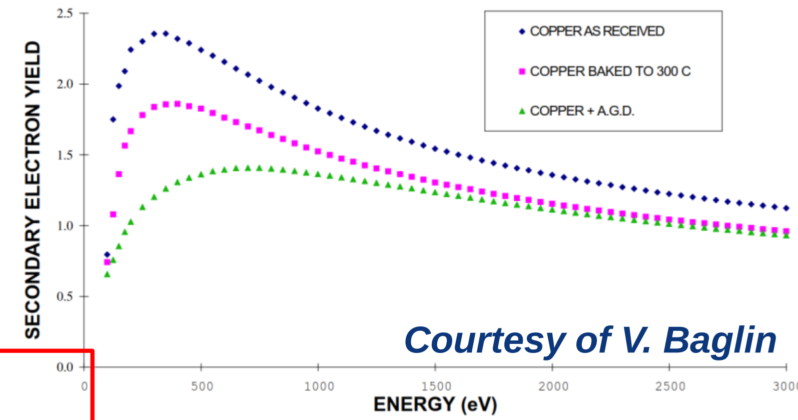
Material	Q_s [mbar·l/ s·cm²]	Q_{jaws} [mbar·l/ s]
CuCD + cladding	$7.4 \cdot 10^{-12}$	$2.9 \cdot 10^{-8}$ ☑

Secondary Electron Yield

Graphite



Copper

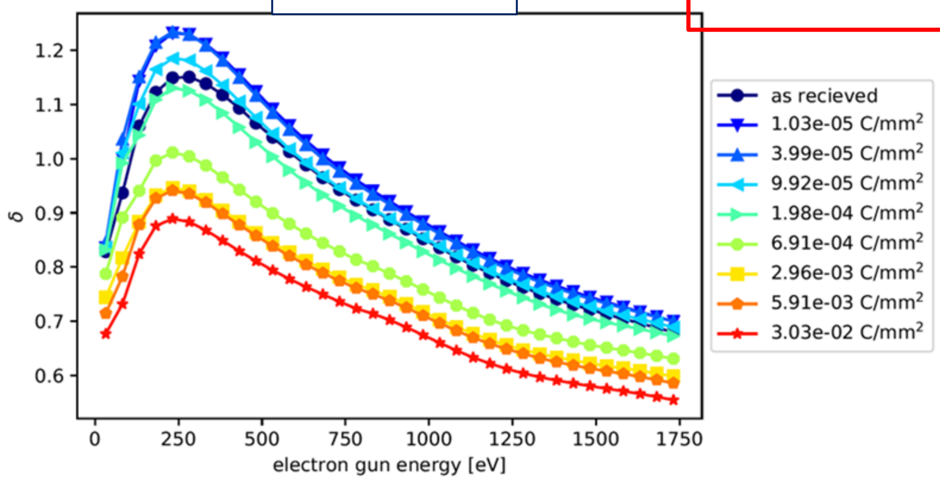


Courtesy of V. Baglin

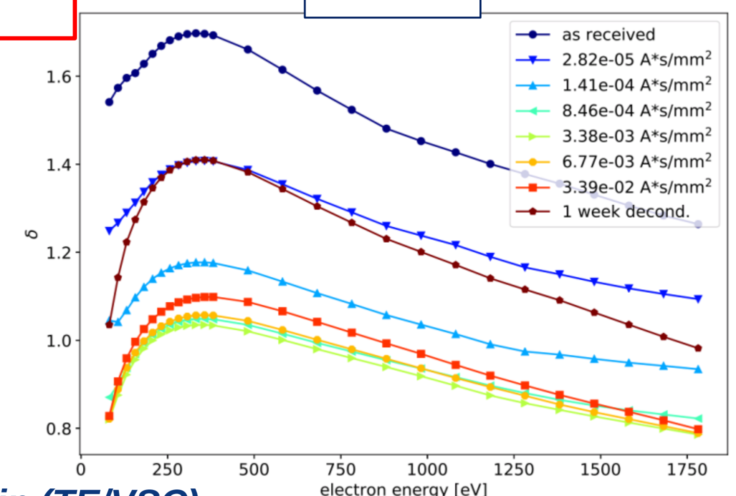
SEY max

collimator~ 1.4

MoGr



CuCD



Courtesy of D. Zanin (TE/VSC)

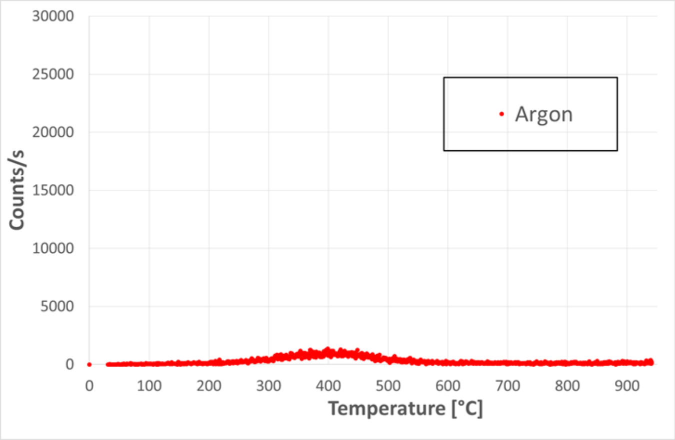
Conclusions and outlook

- A **complete** characterization (outgassing, TDS, microscopy, knowledge of the production process,..) is required to understand the phenomena
- Air content in MoGr has to be reduced during the **production**:
  - higher T (Reproducible? Why?)
  - under vacuum compaction
  - longer annealing
- CuCD: promising material, air exposure to be checked
- **Coating** characterization:
  - surface preparations effect on outgassing ✓
  - coating effect on outgassing (ongoing)
  - adhesion

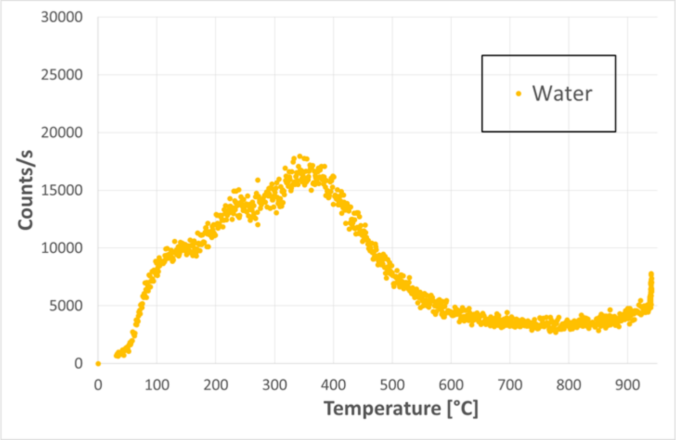
ENGINEERING DEPARTMENT

Thank you for the attention!

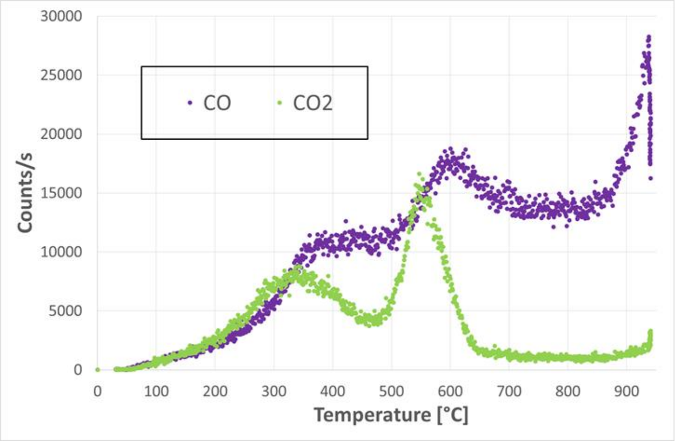
TDS-MoGr



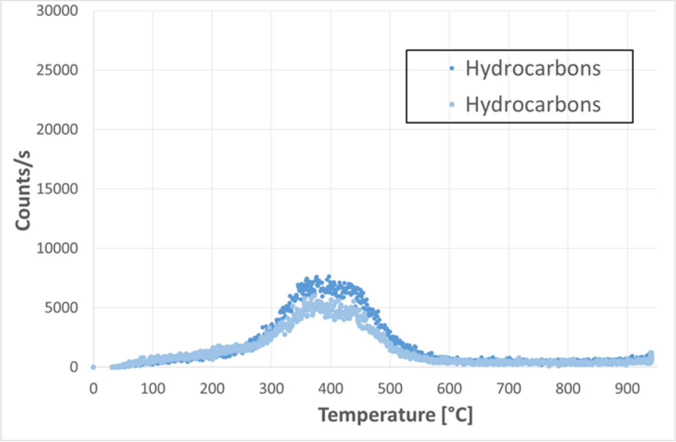
Argon
Counts/s
30000
25000
20000
15000
10000
5000
0
0 100 200 300 400 500 600 700 800 900
Temperature [°C]



Water
Counts/s
30000
25000
20000
15000
10000
5000
0
0 100 200 300 400 500 600 700 800 900
Temperature [°C]

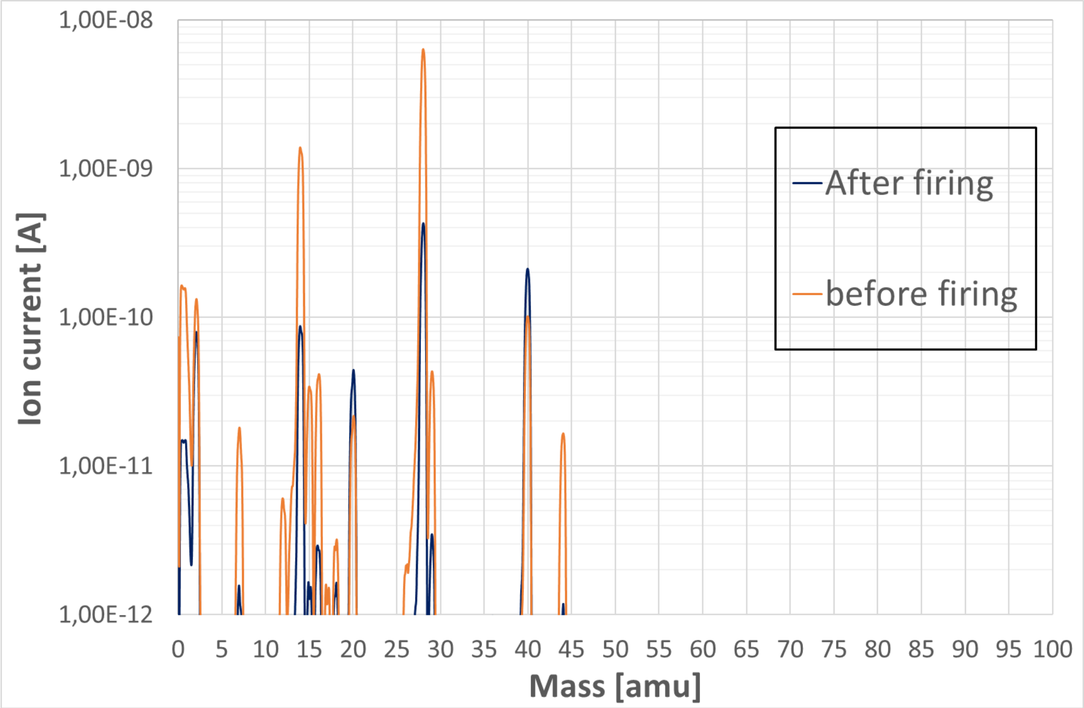


CO
CO2
Counts/s
30000
25000
20000
15000
10000
5000
0
0 100 200 300 400 500 600 700 800 900
Temperature [°C]



Hydrocarbons
Hydrocarbons
Counts/s
30000
25000
20000
15000
10000
5000
0
0 100 200 300 400 500 600 700 800 900
Temperature [°C]

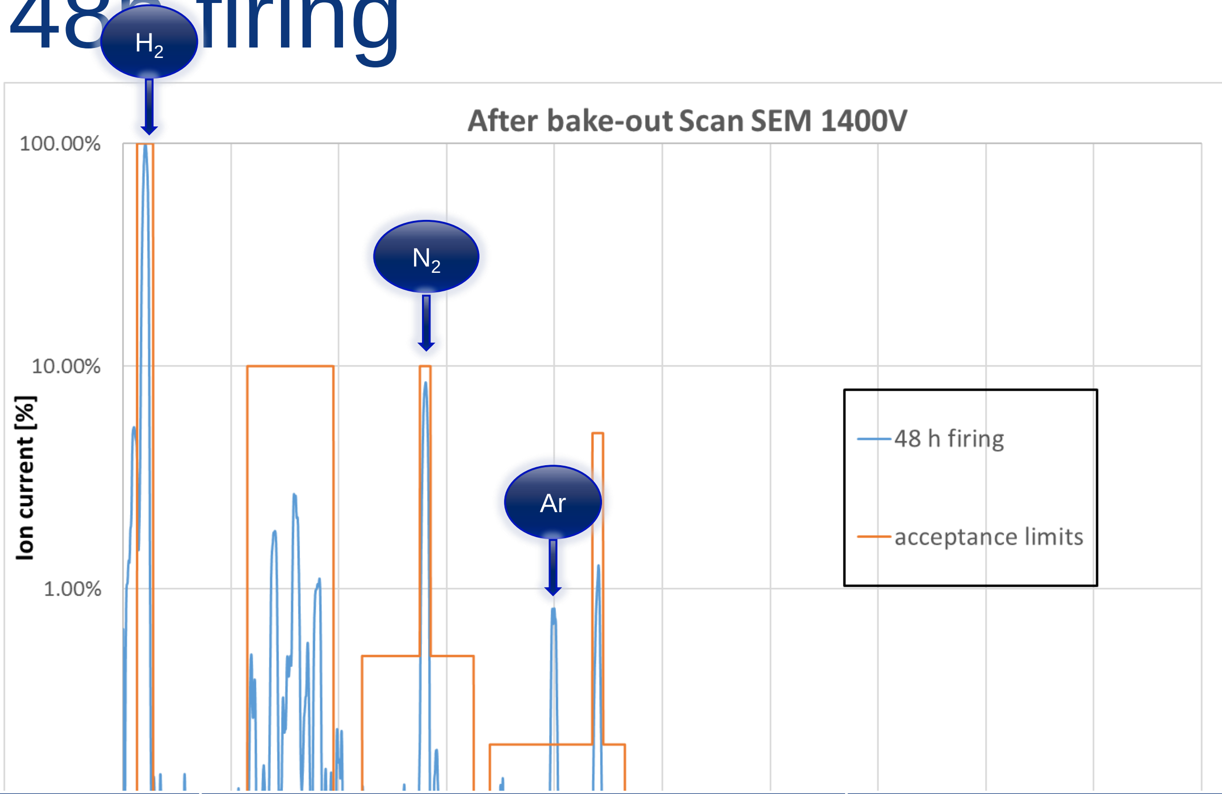
MoGr- 48h firing



48 h, 950°C vacuum firing:

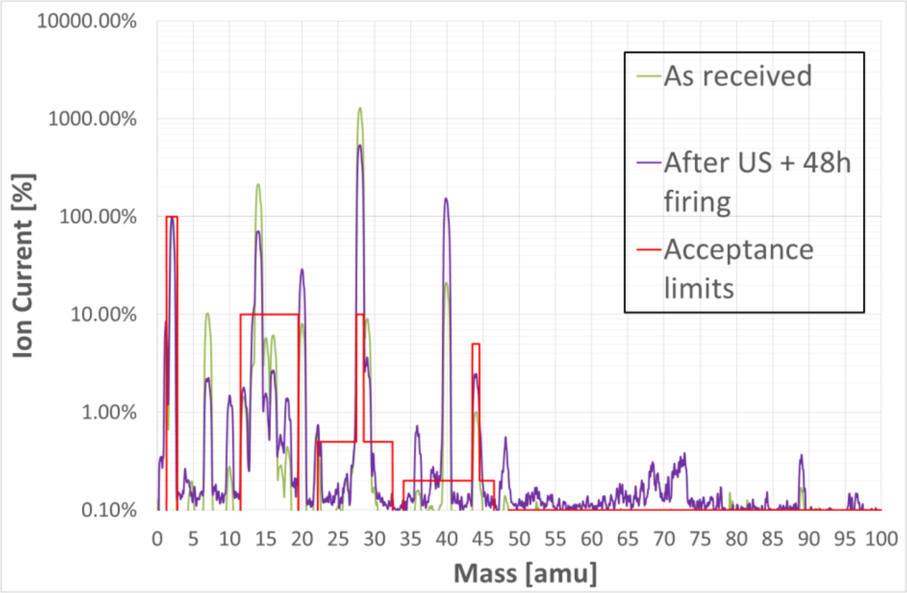
- Q_{before}/Q_{after}= 7, but still out of the limits (~2.5 for 2h firing)
- H_2 decreased
- N_2 decreased, Ar not

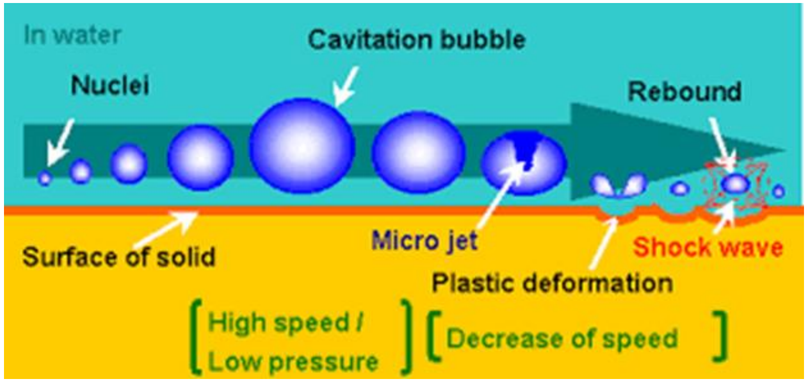
CFC- 48h firing



Treatment	Total outgassing [mbar*l/s]	Internal leak [mbar*l/s]
None	$7.5 \cdot 10^{-8}$ ☒	$8.3 \cdot 10^{-9}$ ☒
2h firing, 950°C	$1.1 \cdot 10^{-8}$ ☑	$5.7 \cdot 10^{-9}$ ☒
48h firing, 950°C	$6.9 \cdot 10^{-9}$ ☑	$3.5 \cdot 10^{-10}$ ☑

US cleaning-first supplier



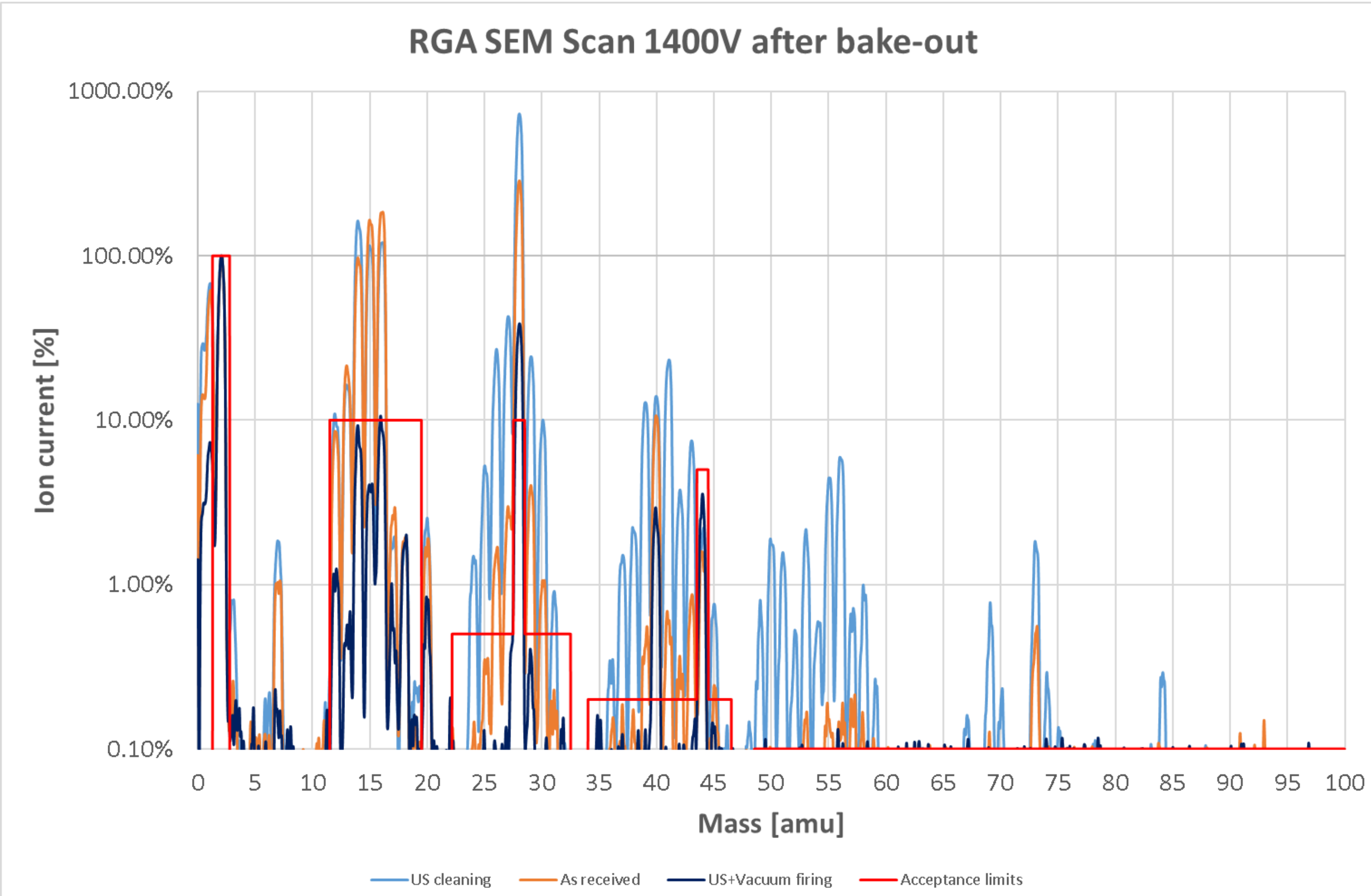


US cleaning + 48 h firing:

- Q_{before}/Q_{after}= 16
- N_2 decreased, Ar constant (normalized graph)

→ result similar to just 48 h vacuum firing

US cleaning-last grade



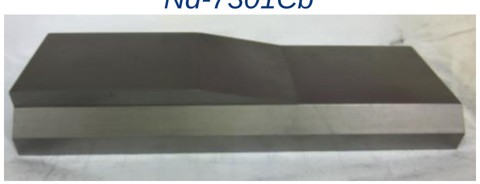
Outgassing and thermal treatment

Grade	Thermal treatment	Total outgassing jaws [mbar*l/s]	Internal leak [mbar*l/s]	Gas analysis
Nd-7301Cb	VF 950°C 48h, Ar venting	$1 \cdot 10^{-8}$ ☑		☒
Nd-7301Cb	VF 950°C 48h, Ne venting	$2.5 \cdot 10^{-9}$ ☑	$2.2 \cdot 10^{-9}$ ☑	☑
Nw-8301Ed	None	$1.8 \cdot 10^{-7}$ ☒	$9.3 \cdot 10^{-9}$ ☒	☒
Nw-8301Ed	VF 950°C 48h, air venting	$1.4 \cdot 10^{-8}$ ☑	$5.3 \cdot 10^{-9}$ ☑	☑

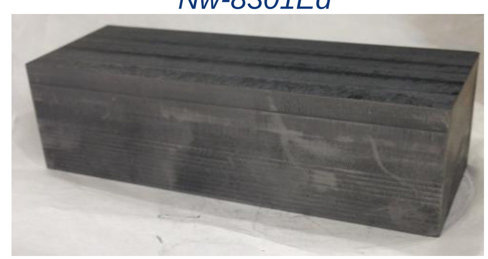
Reduction ~10

Reduction ~1.8

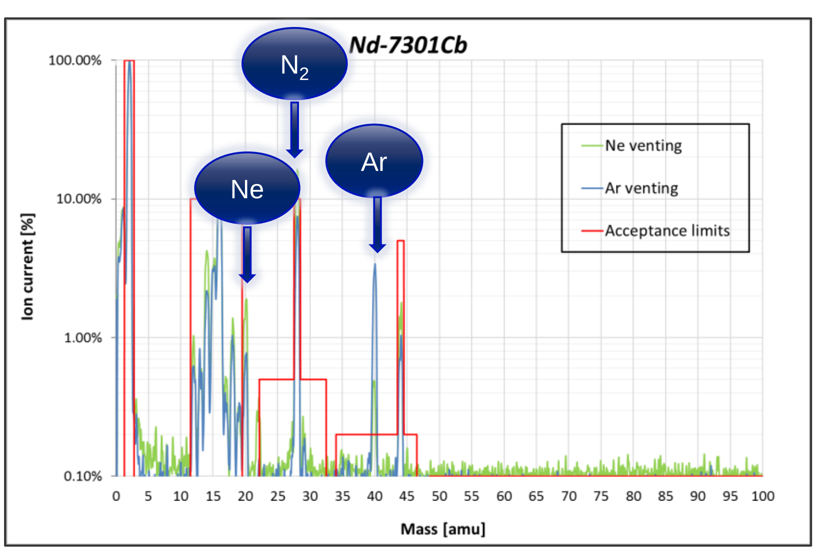
Nd-7301Cb

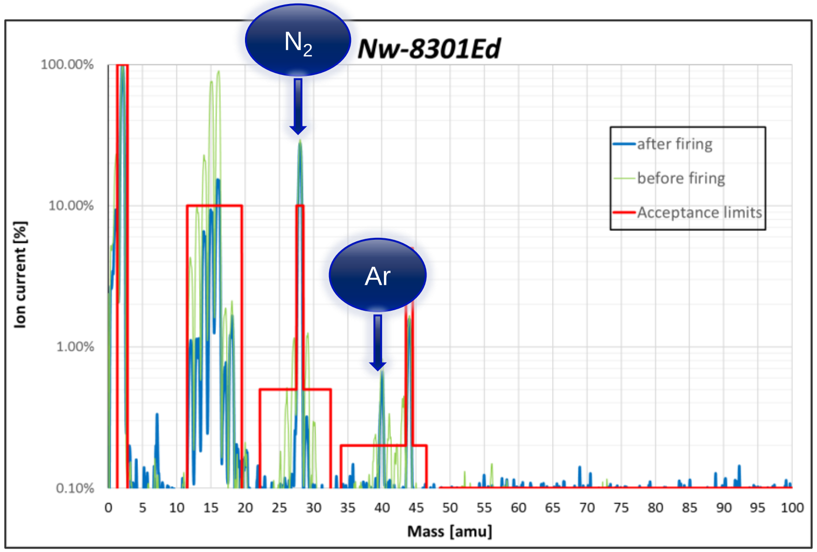


Nw-8301Ed



Gas venting after firing 48h



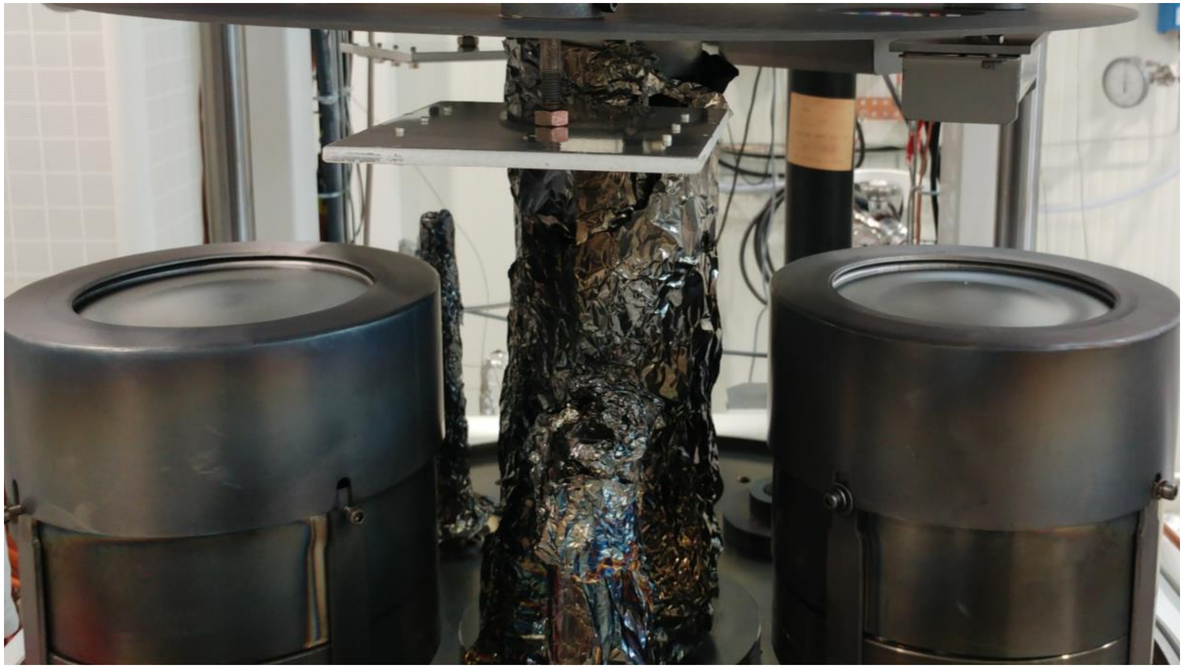


- H_2 dominated
- Adsorption of venting gas

- H_2 dominated
- CH_4 reduction

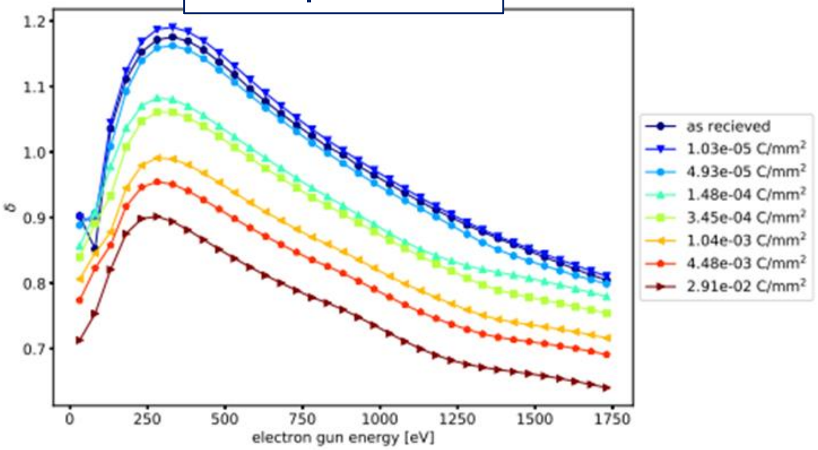
Low outgassing + H2 dominated spectra + Low internal leak → Compliant after 48h firing

Mo coating machine



Secondary Electron Yield

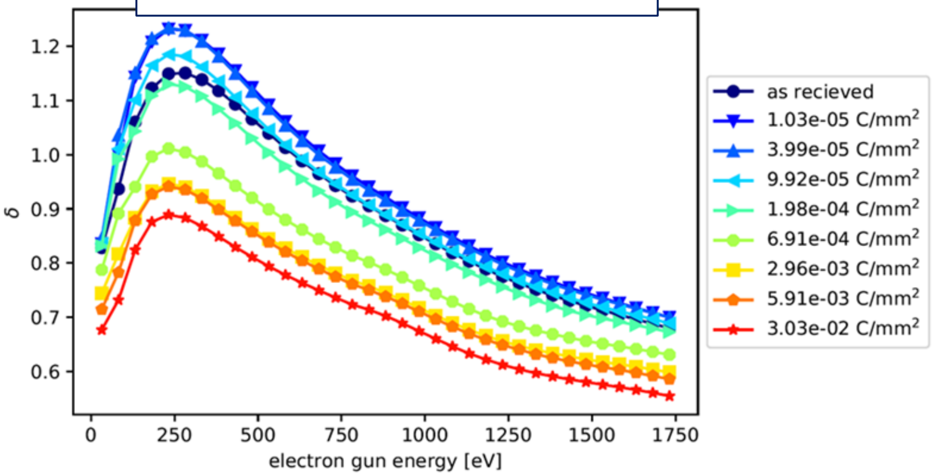
Graphite ☑



SEY max

collimator~ 1.4

MoGr as received ☑



MoGr air exposure ☑