



Politecnico
di Bari



Chemical analyses of deposits in the RPC chambers

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Overview

- ◎ High Pressure Laminate surface analysis
 - Visual inspection
 - Energy Dispersive X-Ray Spectroscopy
 - Scanning Electron Microscope
- ◎ Hydrogen Fluoride study

Surface analysis motivation

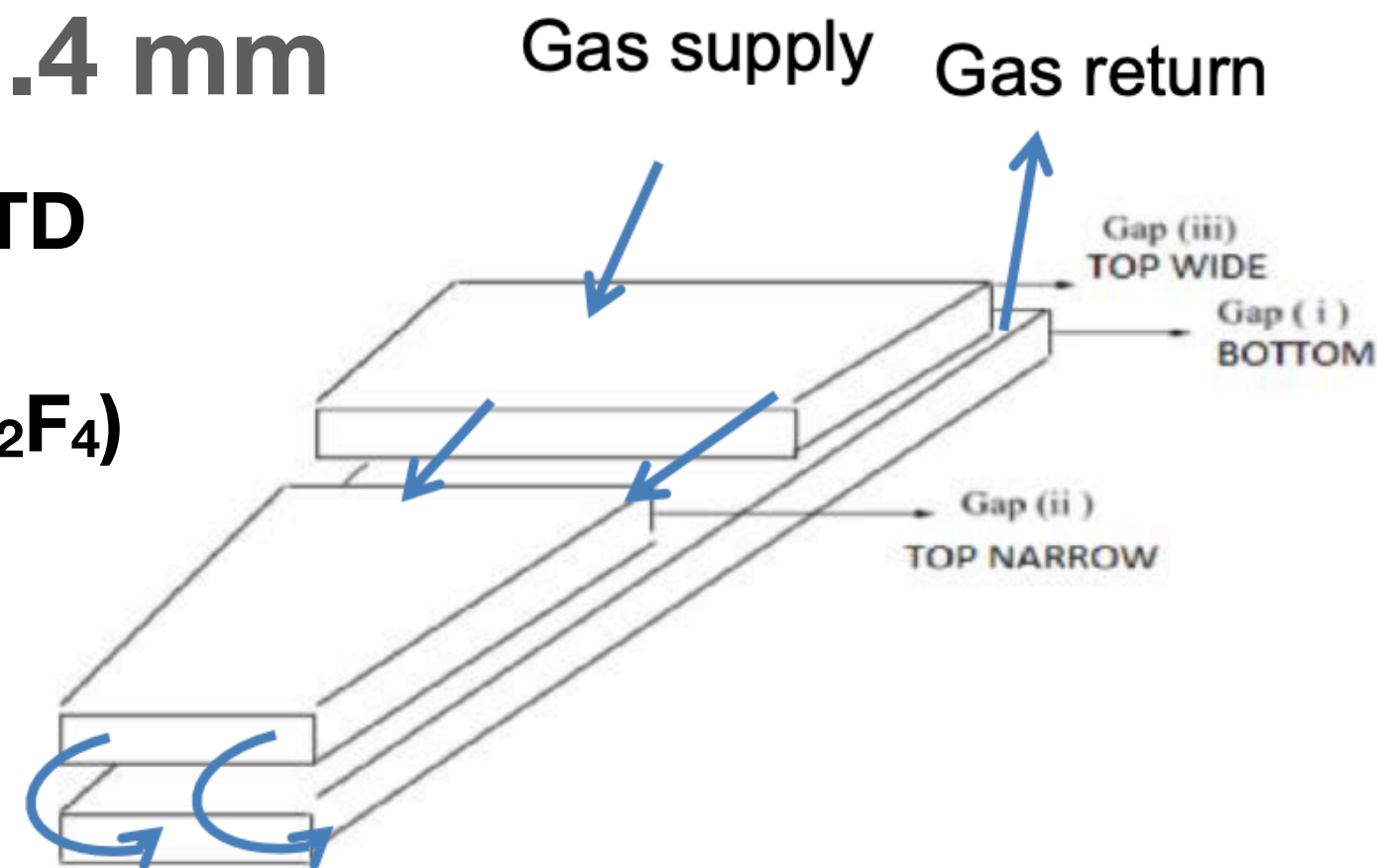
Layout of RPC prototype-1 for CMS Upgrade project (iRPC)

Trapezoidal shape

(h: 161.3, ls: 86.6, ss: 58.4 cm)

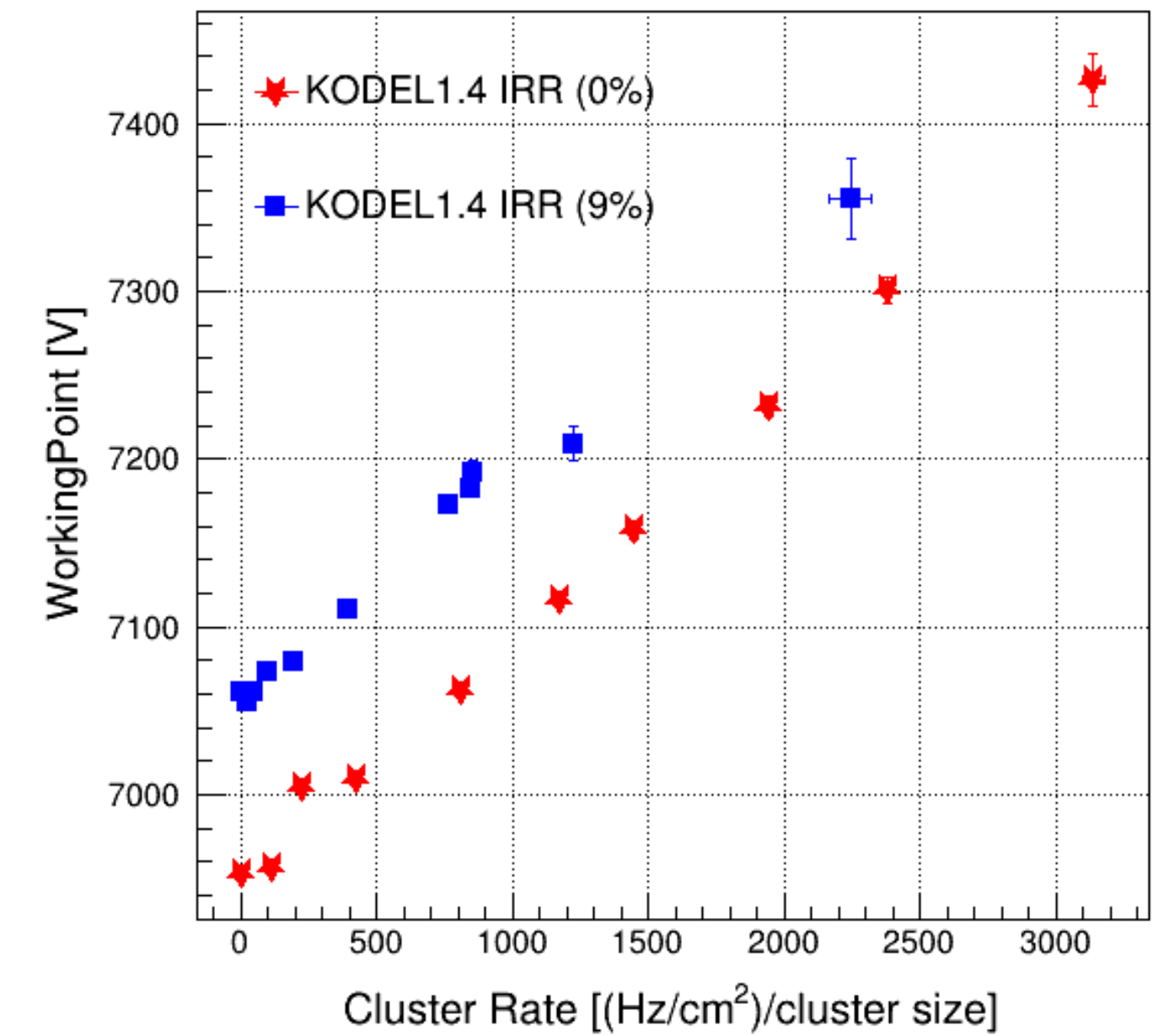
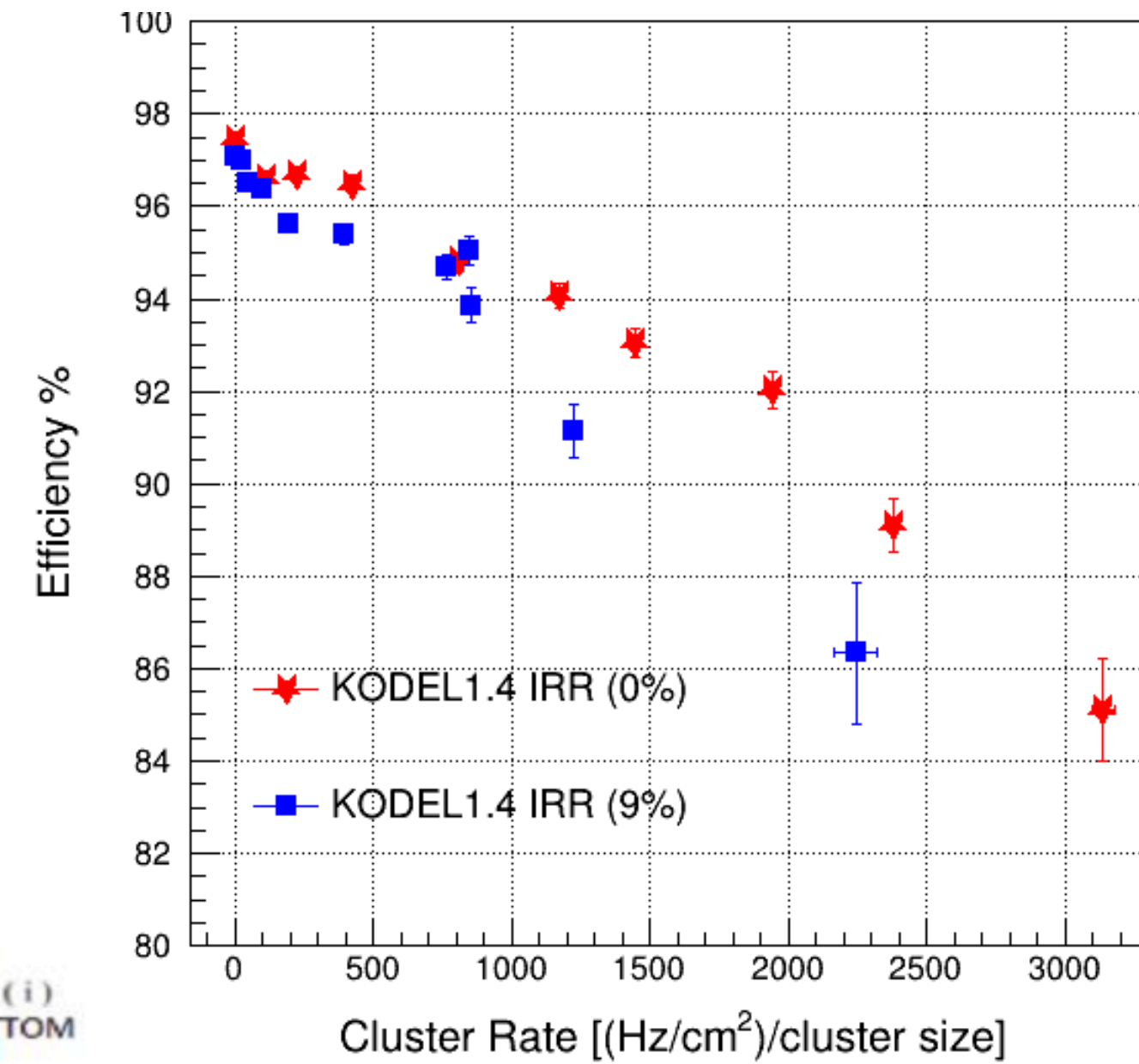
Double gap, HPL thickness: 1.43 mm,
gap thickness: 1.4 mm

Operation with CMS STD
mixture: ~ 95%
tetrafluoroethane ($C_2H_2F_4$)

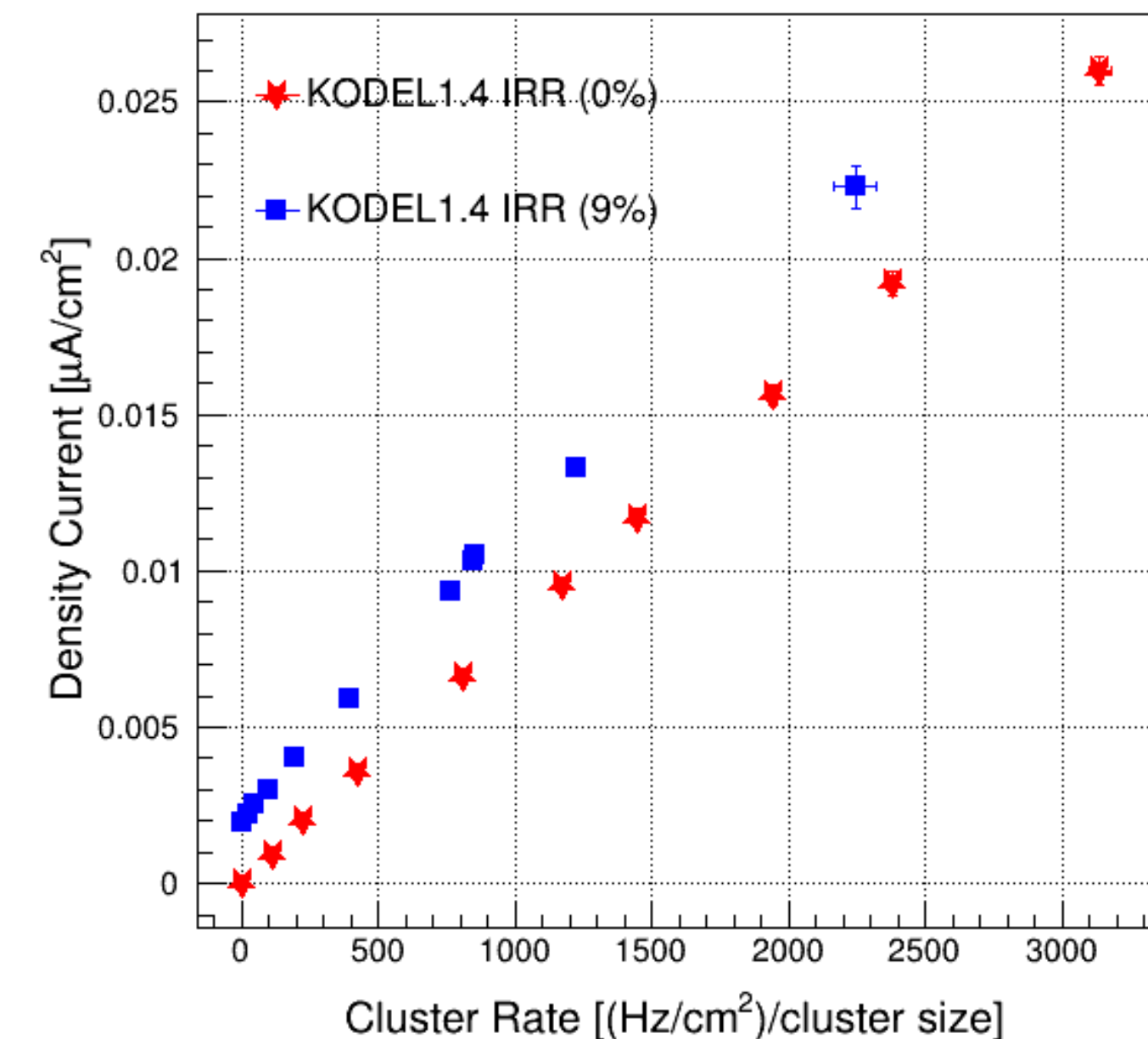


Motivation

- Performance degradation after 90 mC/cm² (August 2018 TB)
- 100V WP shift
- Operational current increase



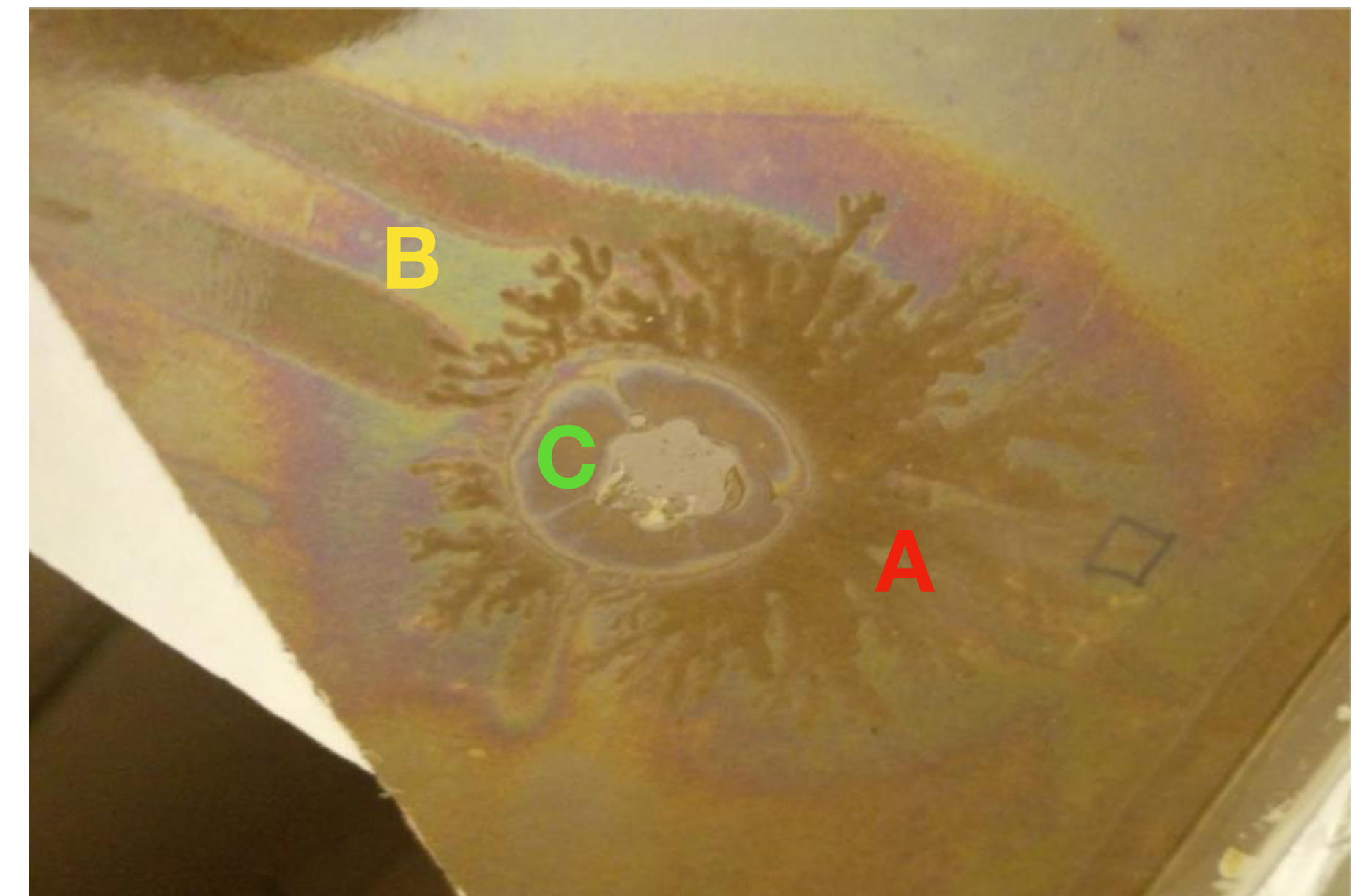
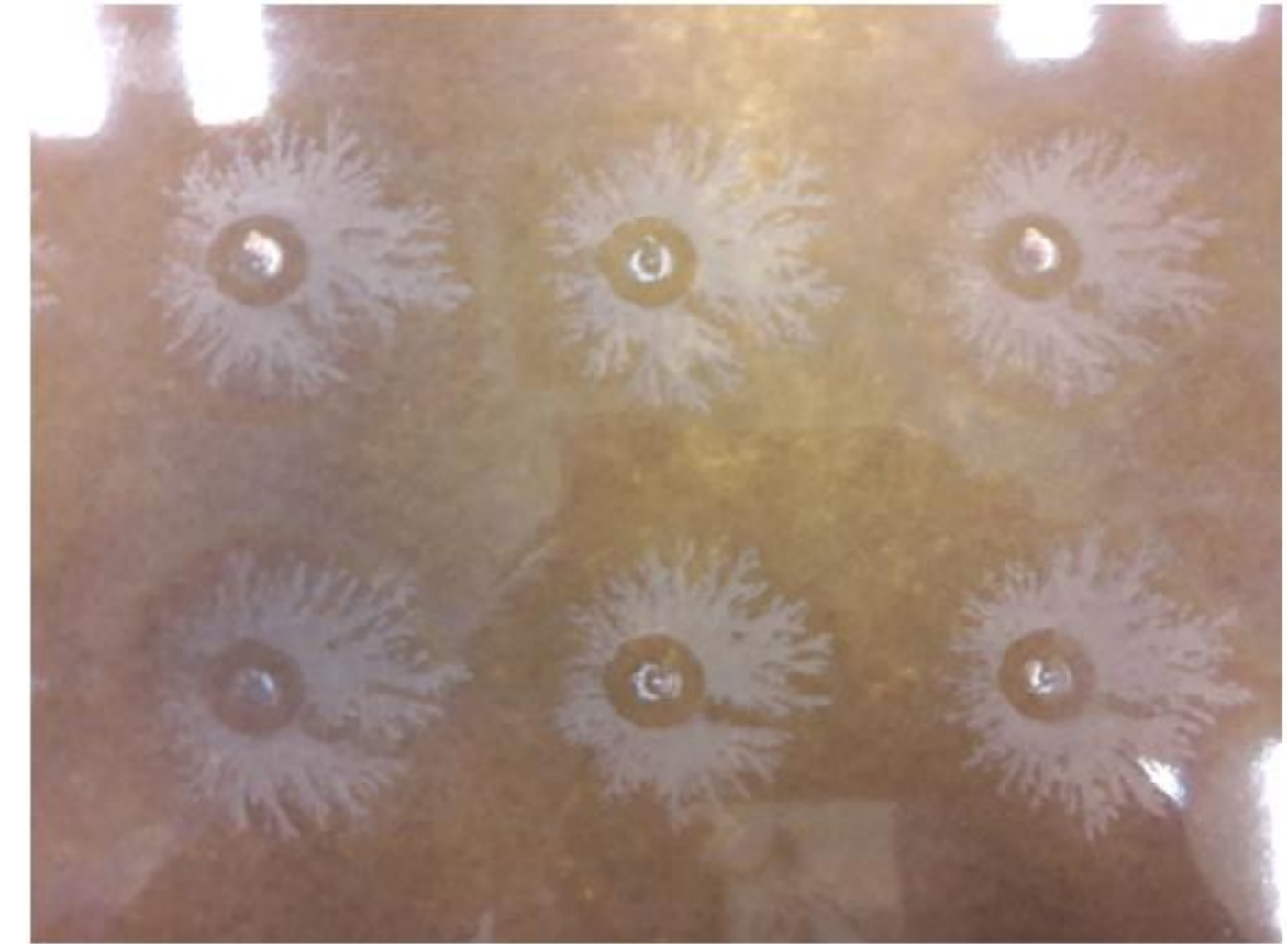
Irradiation campaign @GIF++
Before irradiation (May 2017)
After 9% irradiation target (August 2018)



iRPC visual inspection

Observations

- A.** White matt spot around spacers and oil inlet/outlet
 - B.** Halo near the spacers
 - C.** Ring around spacers and edges look similar to the halo and the below spacers area
- SIMILAR EFFECT OBSERVED IN RE4 IRRADIATED @ GIF++



HPL surface analysis

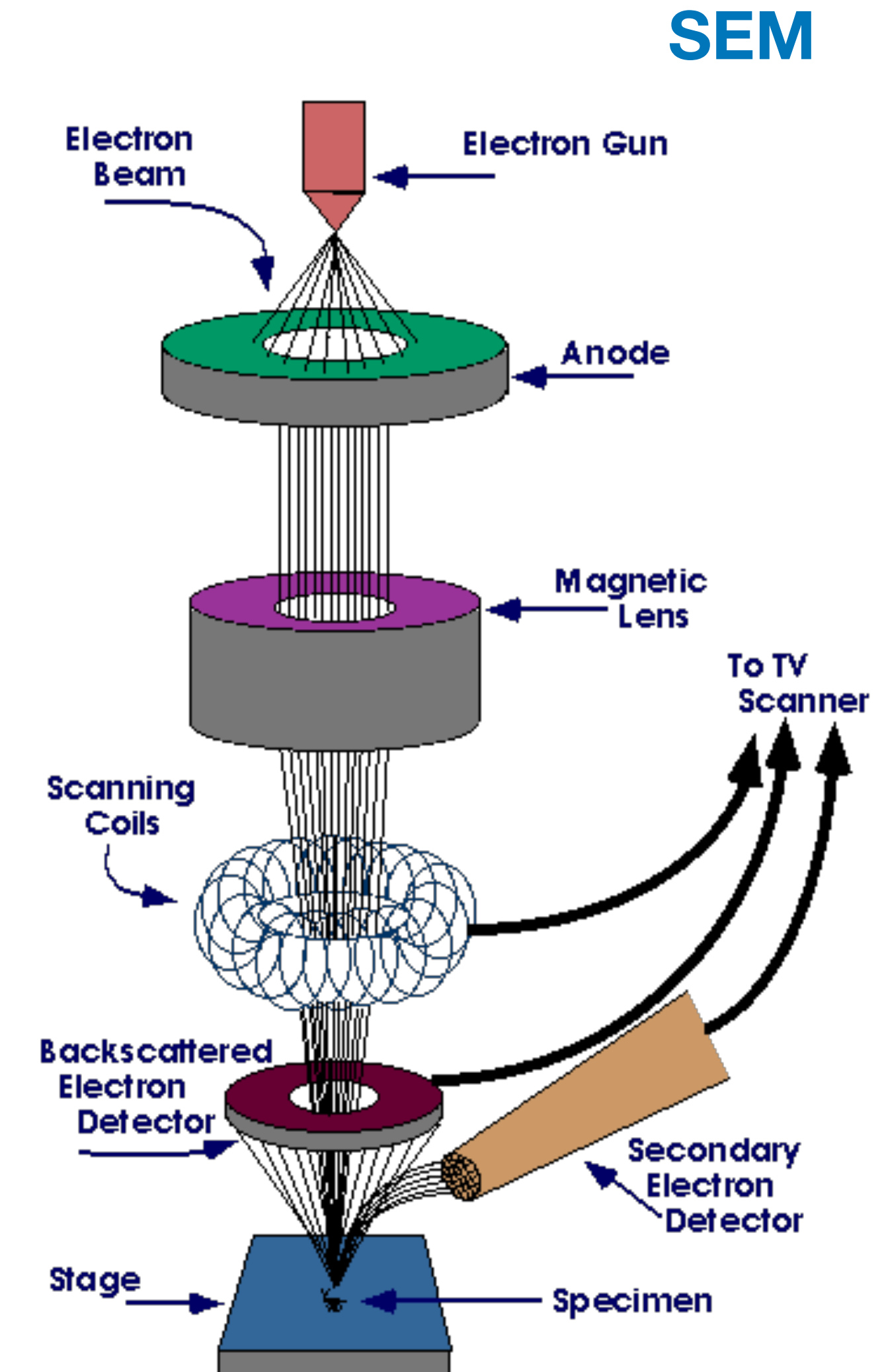
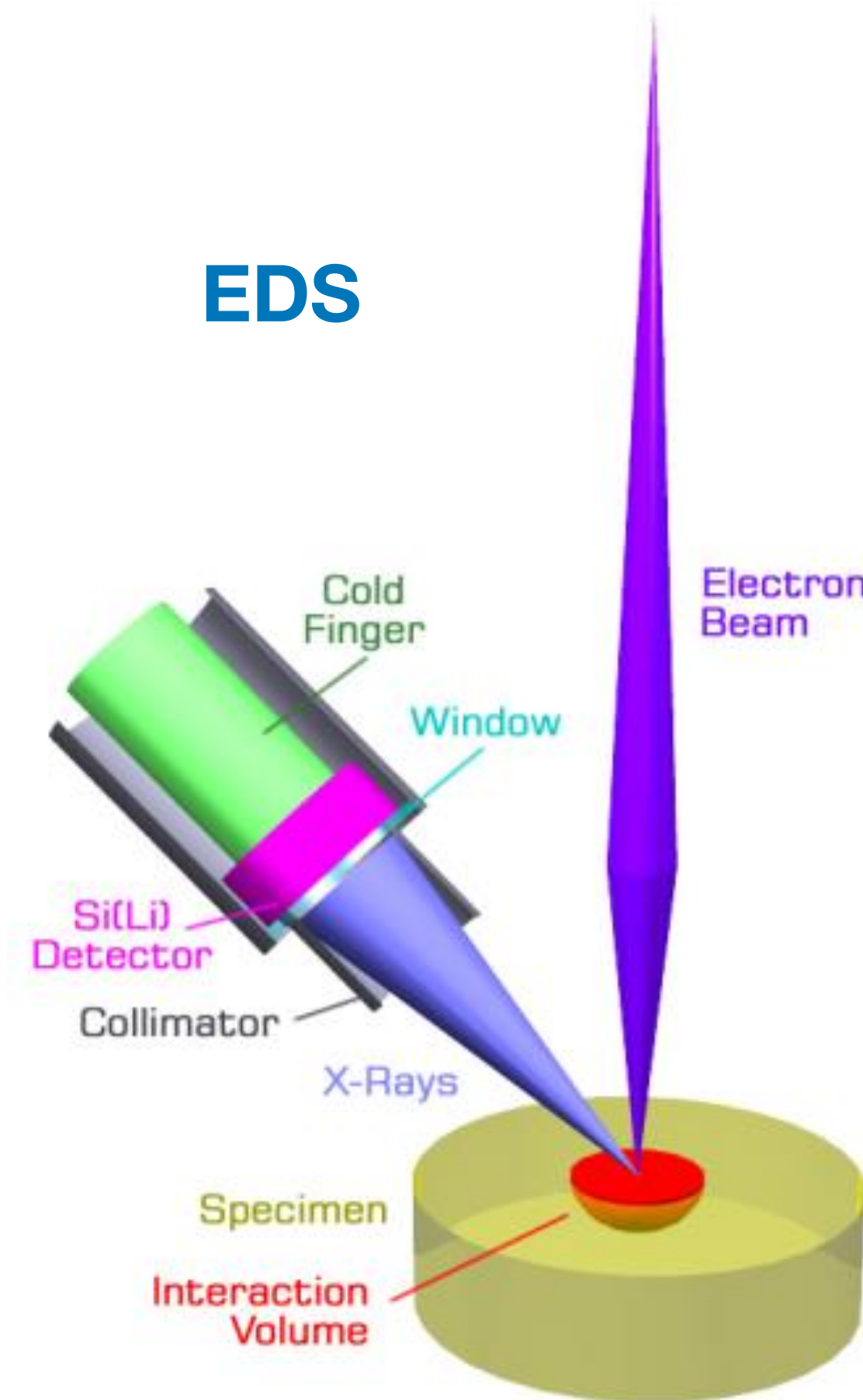
Hypothesis

Bad oil polymerization

Glue reaction

Surface analysis techniques

- EDS (Energy Dispersive X-Ray Spectroscopy) @ chemistry Lab
- SEM (Scanning Electron Microscope) @ chemistry Lab

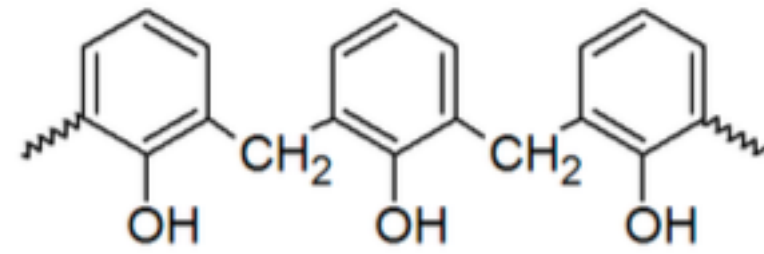


EDS (Energy Dispersive X-Ray Spectroscopy)

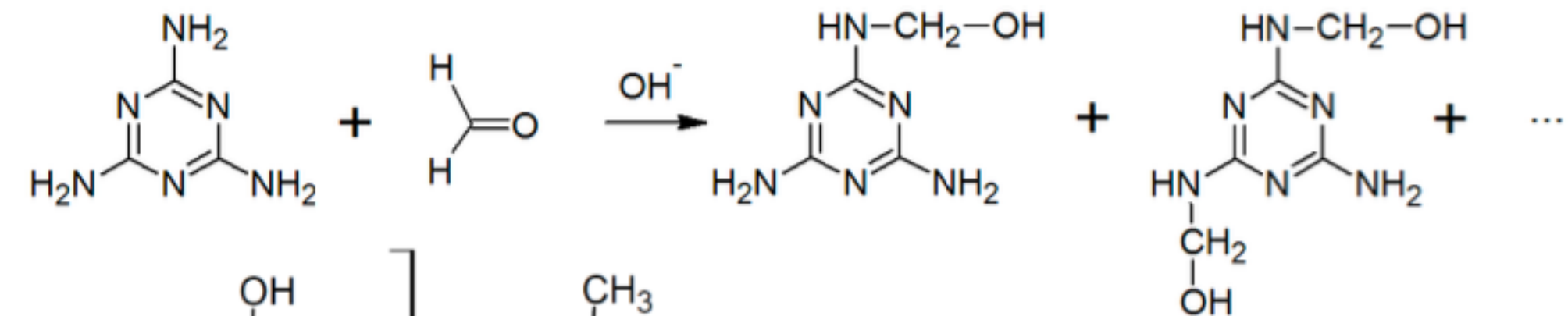
EDS for chemical composition

What to expect?

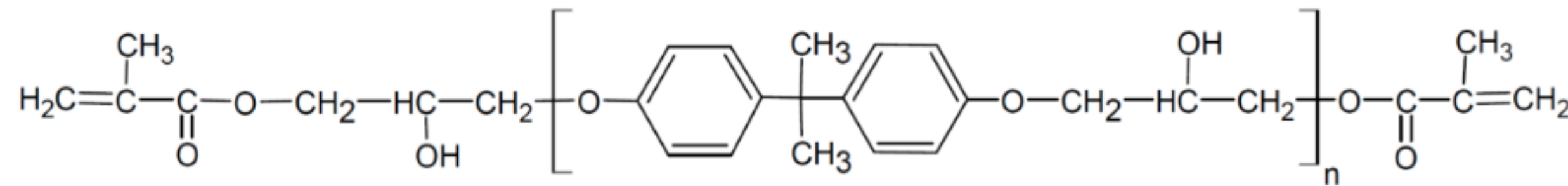
Phenolic resin ((C6-H6-O.C-H2-O)x) = phenol (C6H5OH) + formaldehyde (CH2O)



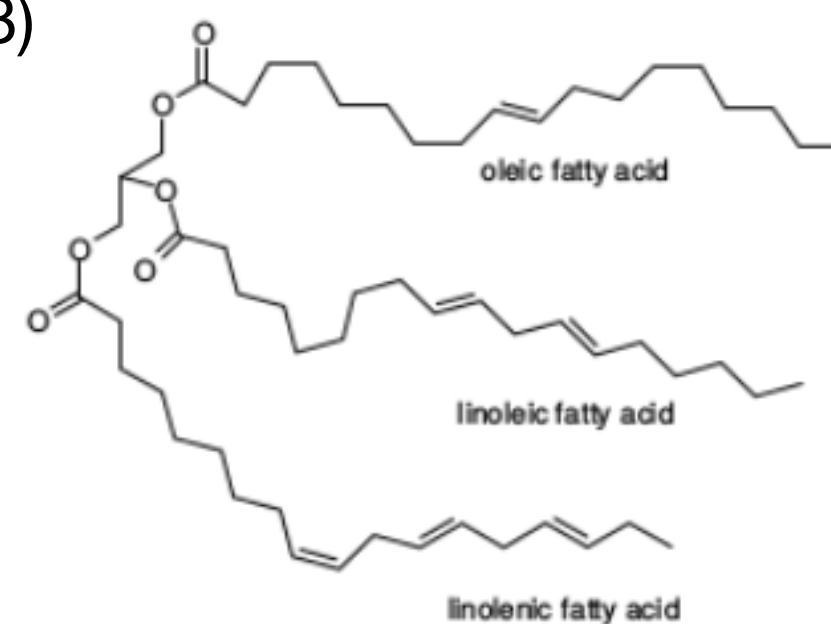
Melamine resin (C4H8N6O) = melamine (C3H6N6) + formaldehyde (CH2O)



Epoxy glue (3M DP460)



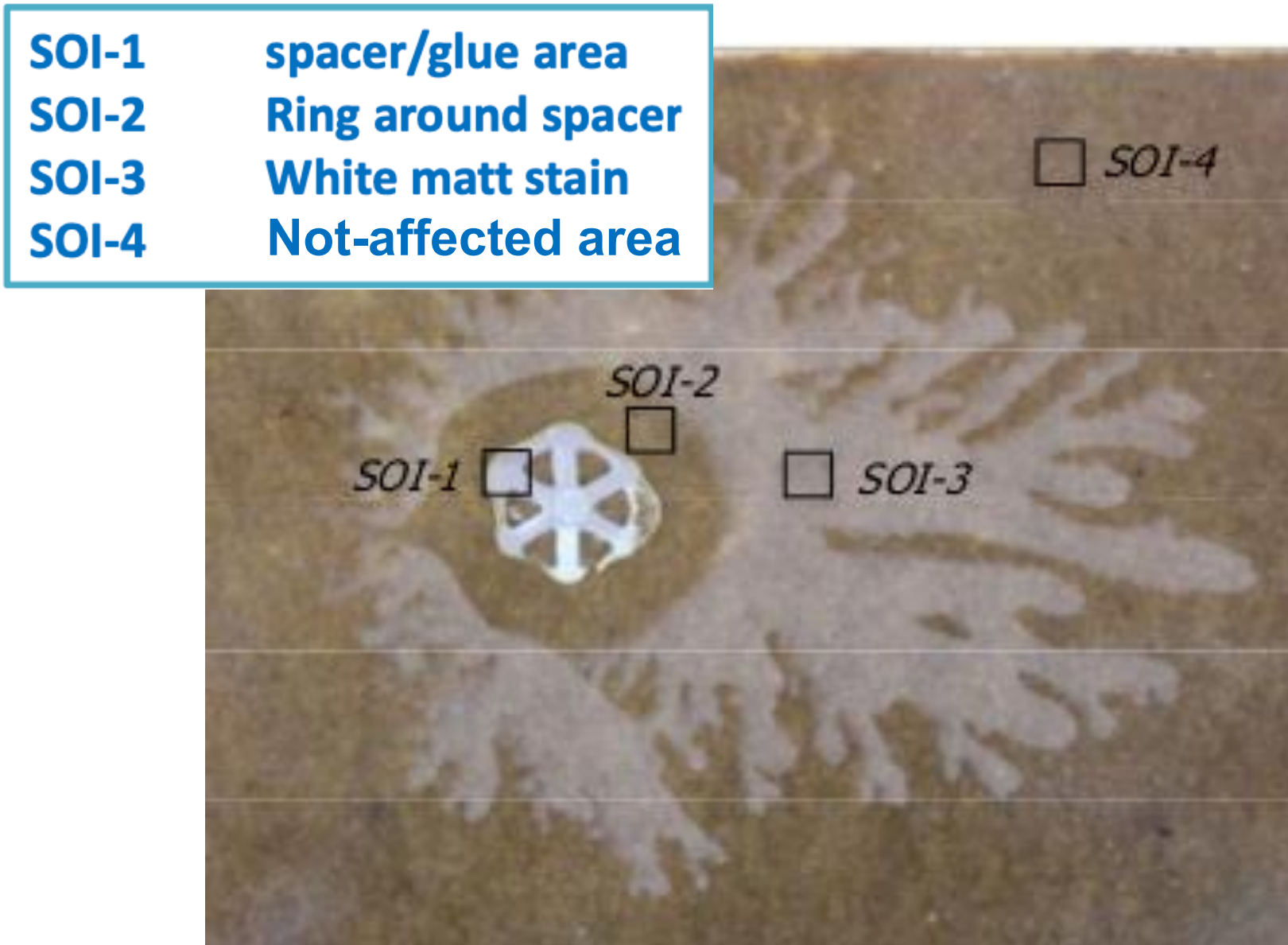
Linseed oil + heptane (CH3(CH2)5CH3)



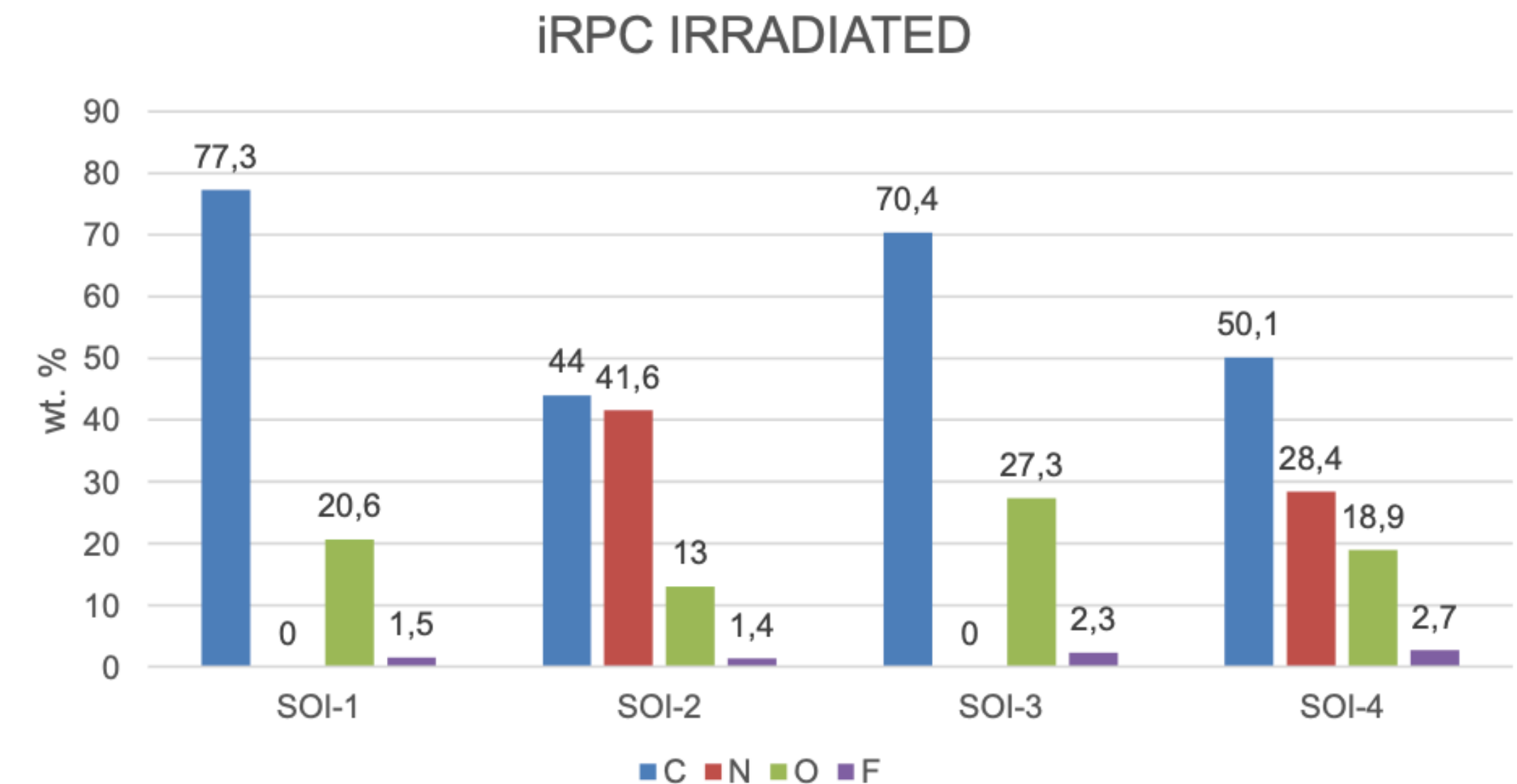
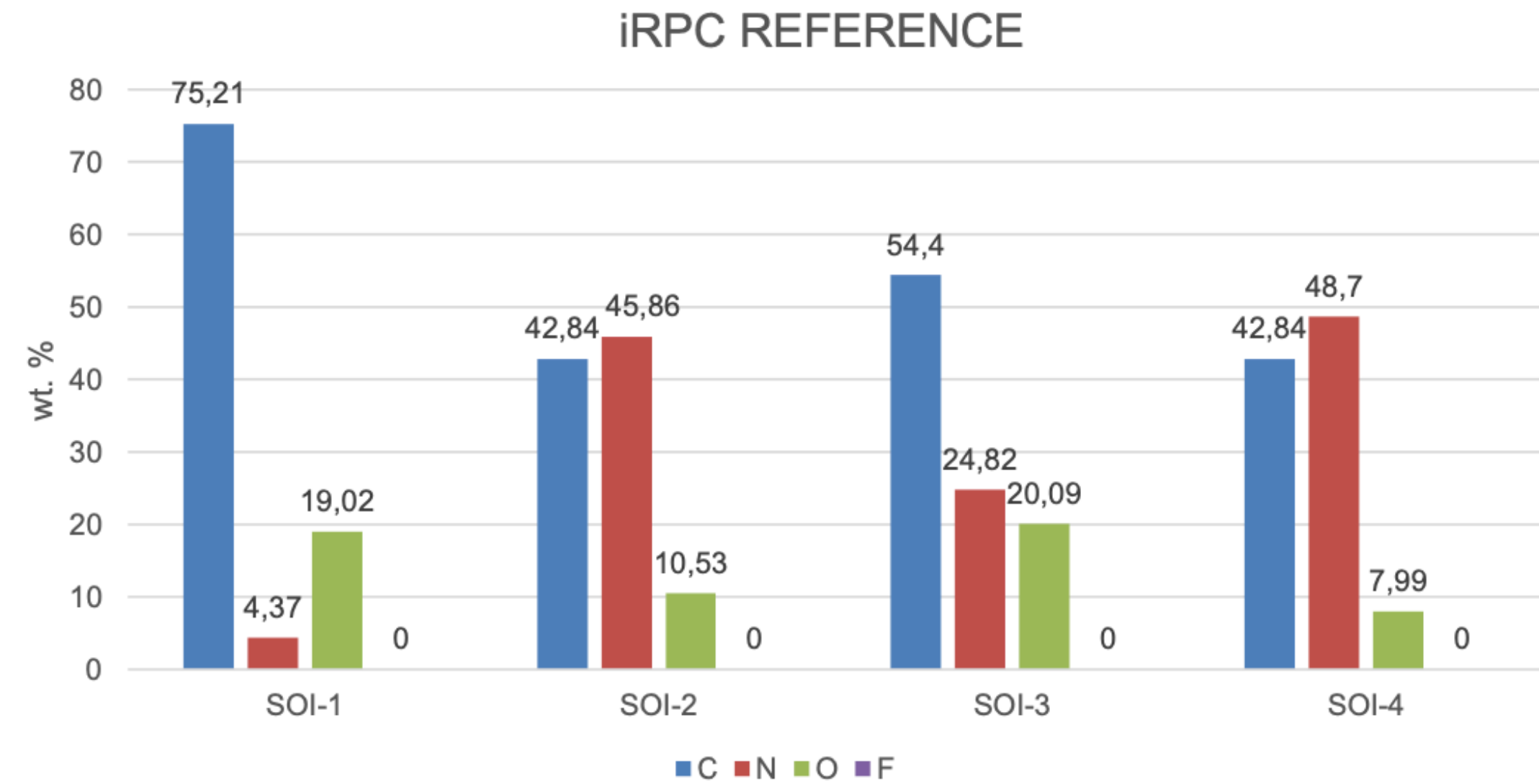
Fatty acid	Formula	Number of double bonds
Stearic	CH ₃ - (CH ₂) ₁₆ - COOH	0
Oleic	CH ₃ - (CH ₂) ₇ - CH = CH - (CH ₂) ₇ - COOH	1
Linoleic	CH ₃ - (CH ₂) ₄ - CH = CH - CH ₂ - CH = CH - (CH ₂) ₇ - COOH	2
Linolenic	CH ₃ - CH ₂ - CH = CH - CH ₂ - CH = CH - CH ₂ - CH = CH - (CH ₂) ₇ - COOH	3

iRPC reference/irradiated

Irradiated chamber view



- Fluorine presence in IRR SOI
- Less N in SOI-3 and 4 IRR
- Similar composition SOI-4 IRR and SOI-3 REF

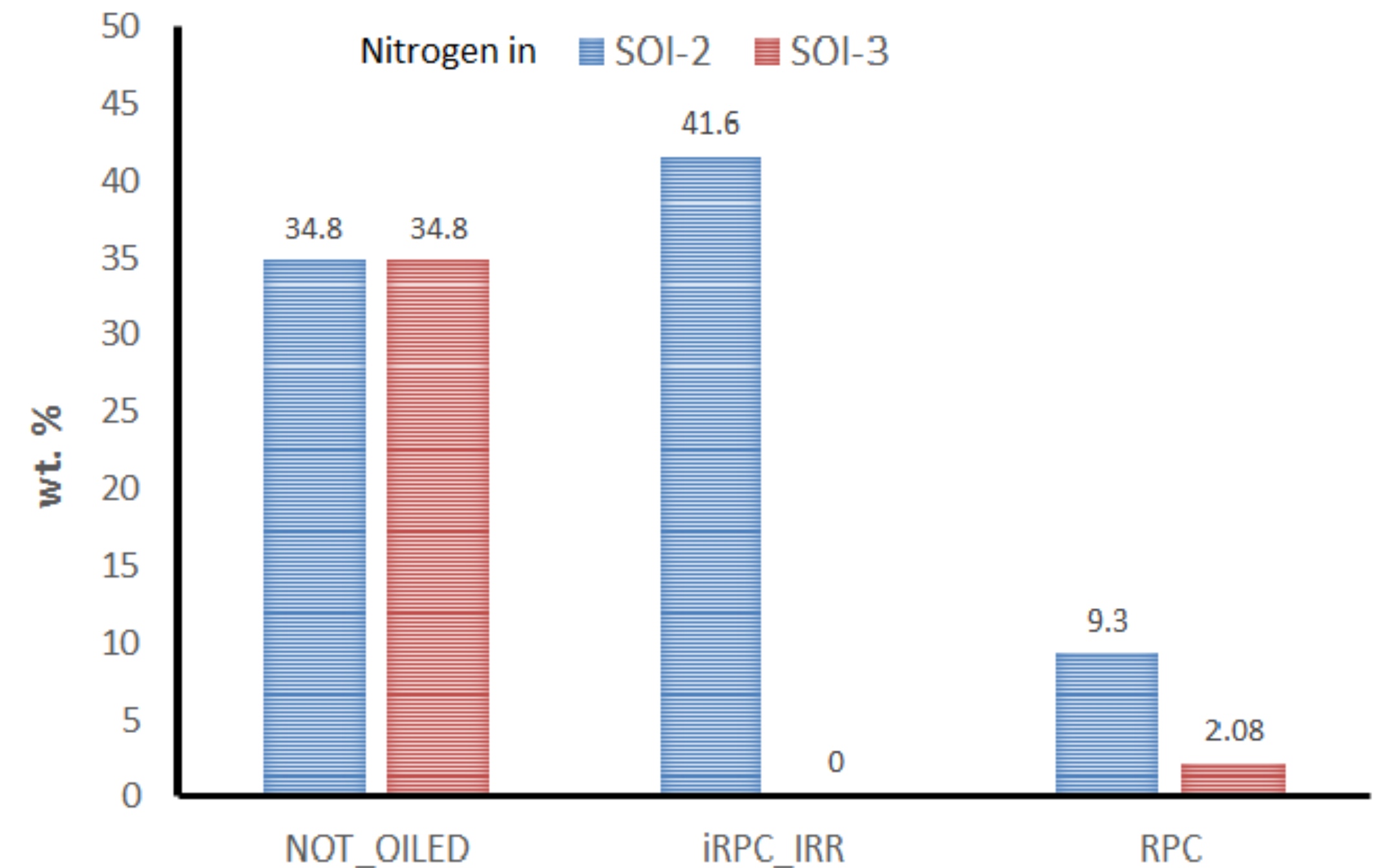


*SOI = site of interest

Nitrogen presence

- N associated the external HPL layer made of melamine resin
- N presence in SOI-2, comparable with the not-oiled sample, indicating a lack of oil
- Thicker oil layer in RPC reference with respect to iRPC_IRR

**SOI-2: Ring around the spacer,
SOI-3: White matt stain**

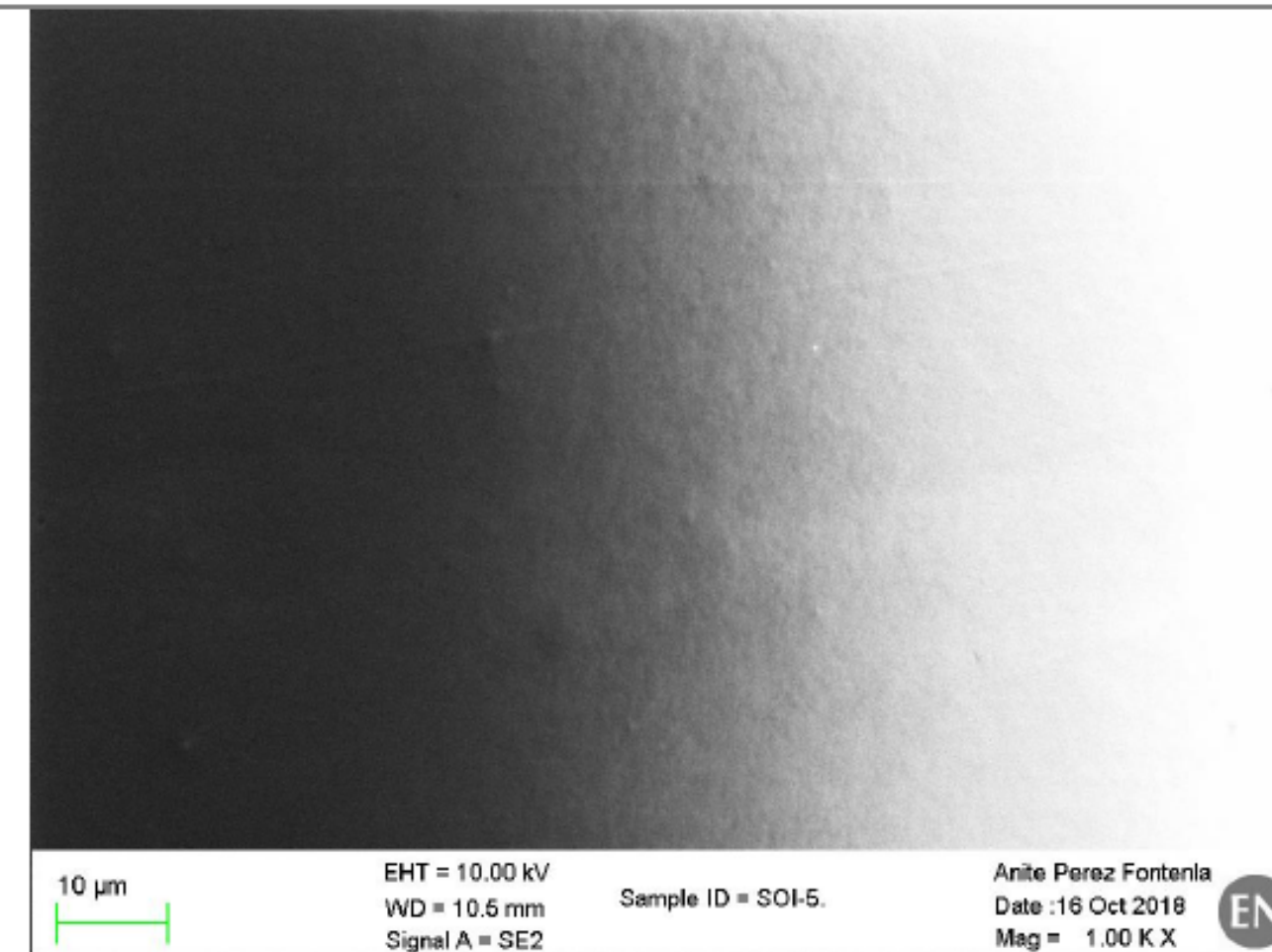


SEM (Scanning Electron Microscope)

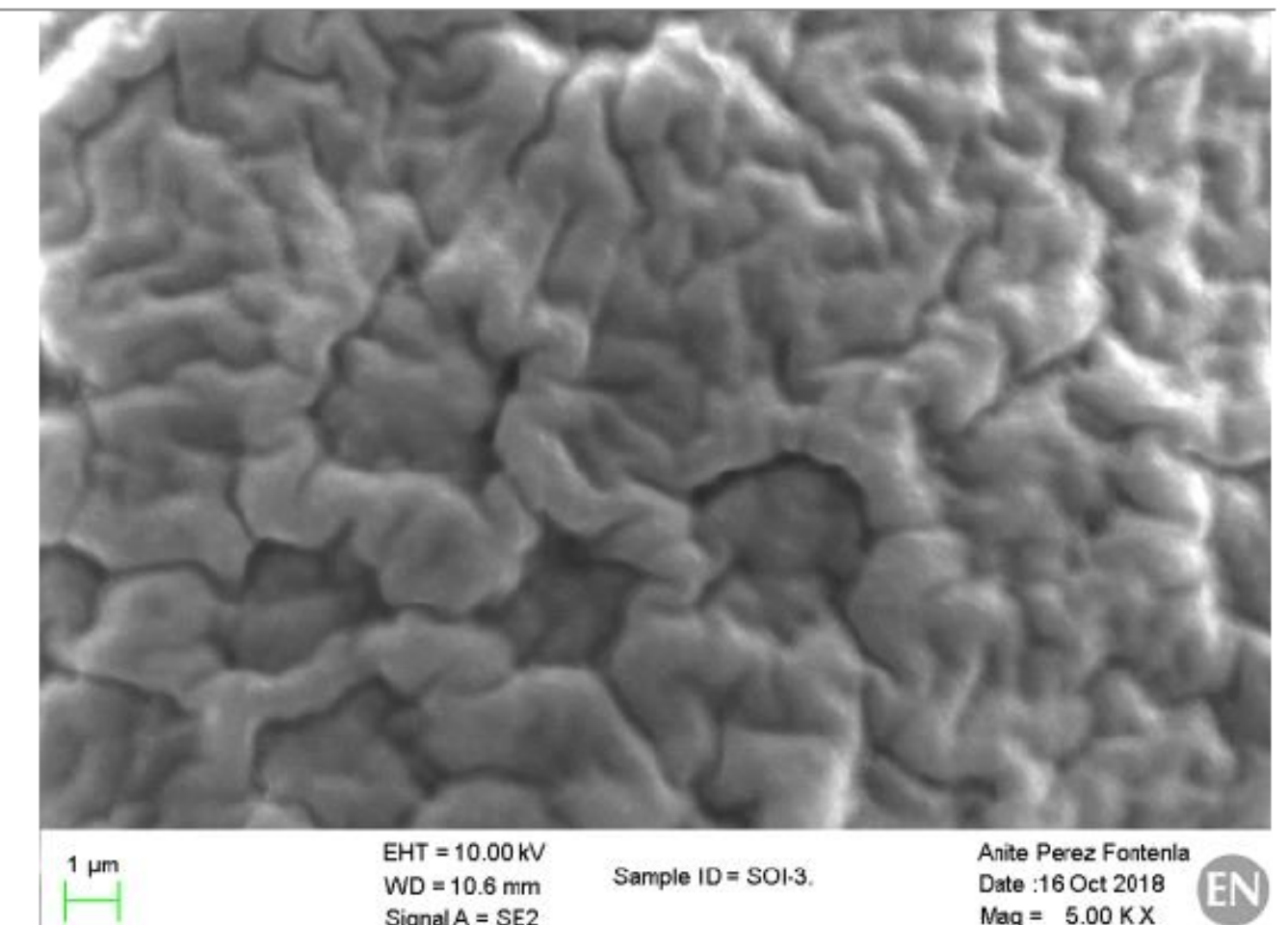
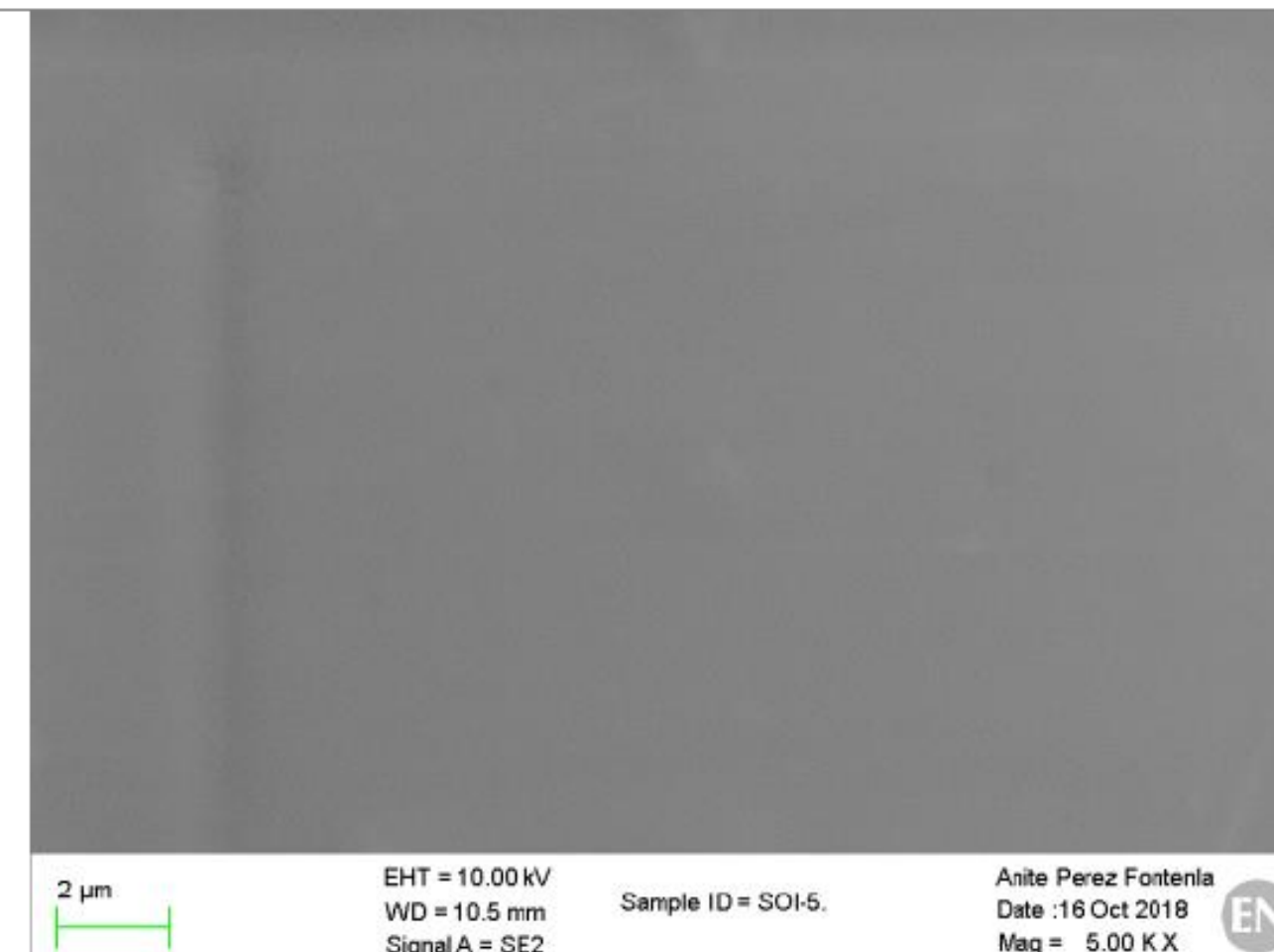
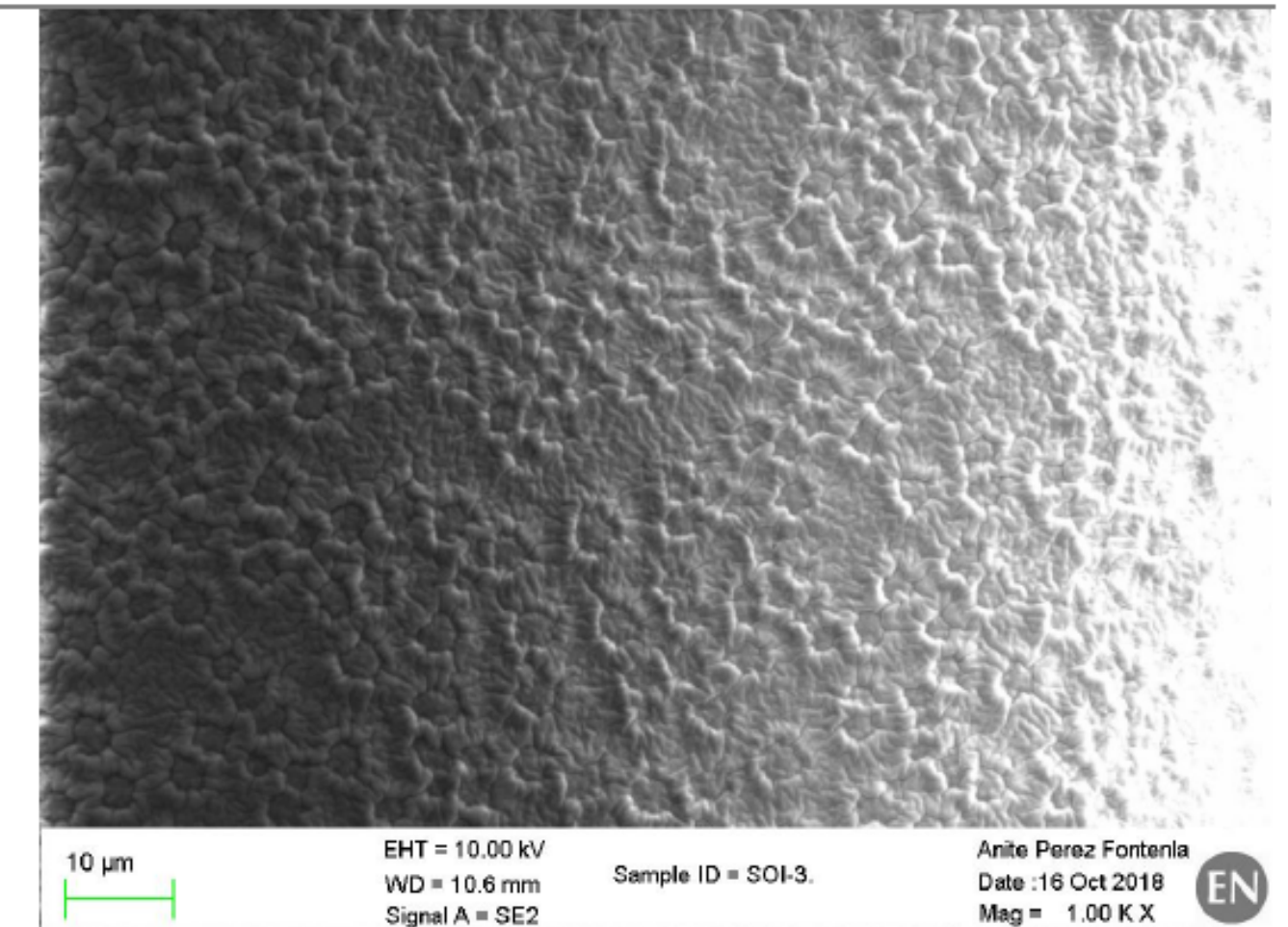
The surface characteristics at the non-affected area and at a randomly selected stain was compared by **SEM imaging on iRPC irradiated sample**, presenting the first one a **smooth aspect** while the second one shows a **rough topography**

iRPC IRRADIATED

Non-affected surface (SOI-4)



Matt stain (SOI-3)

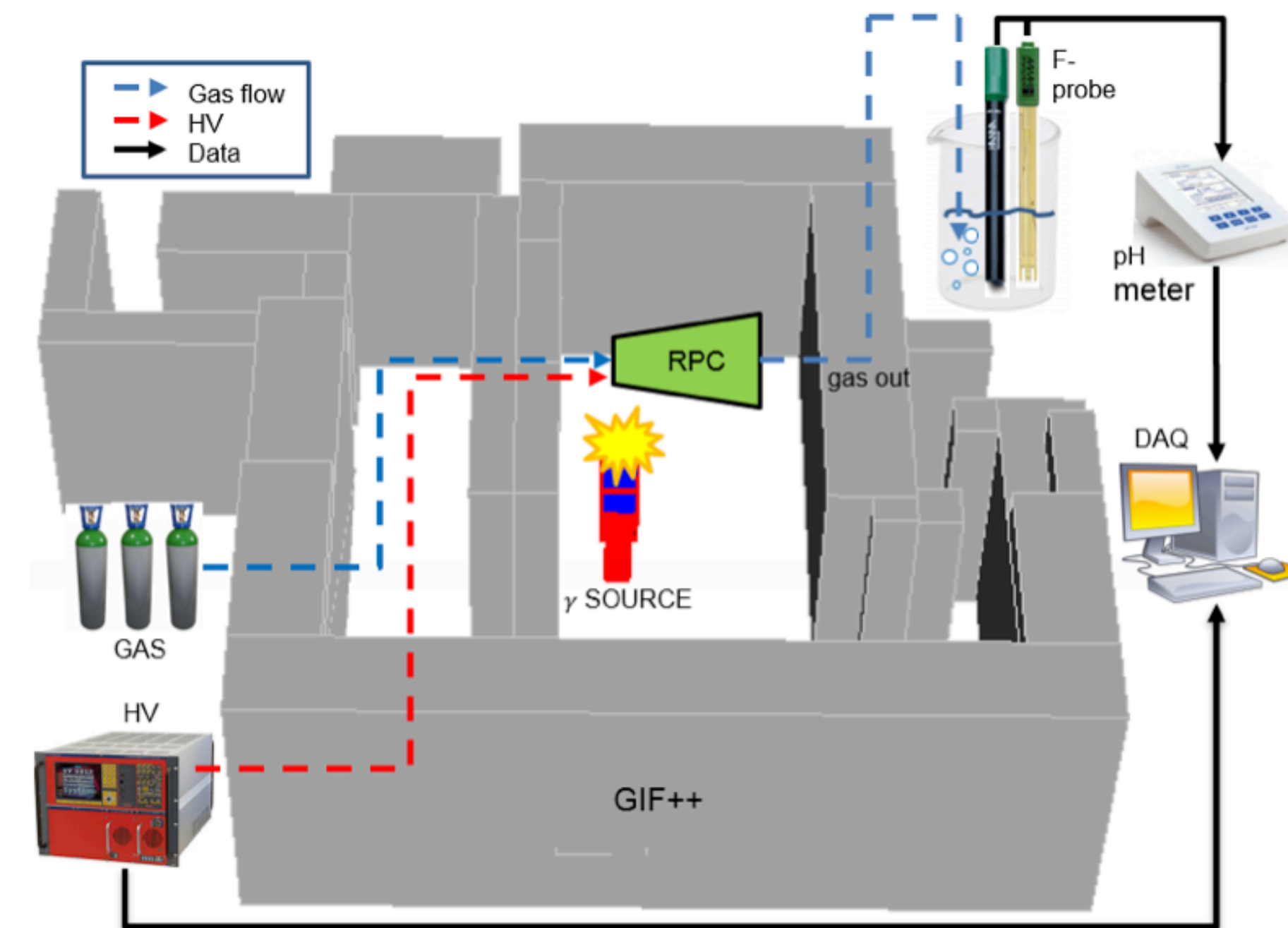


Decomposition of the C₂H₂F₄ molecules, induced by the relatively high-energy photons (i.e. UV) -> F⁻, then:



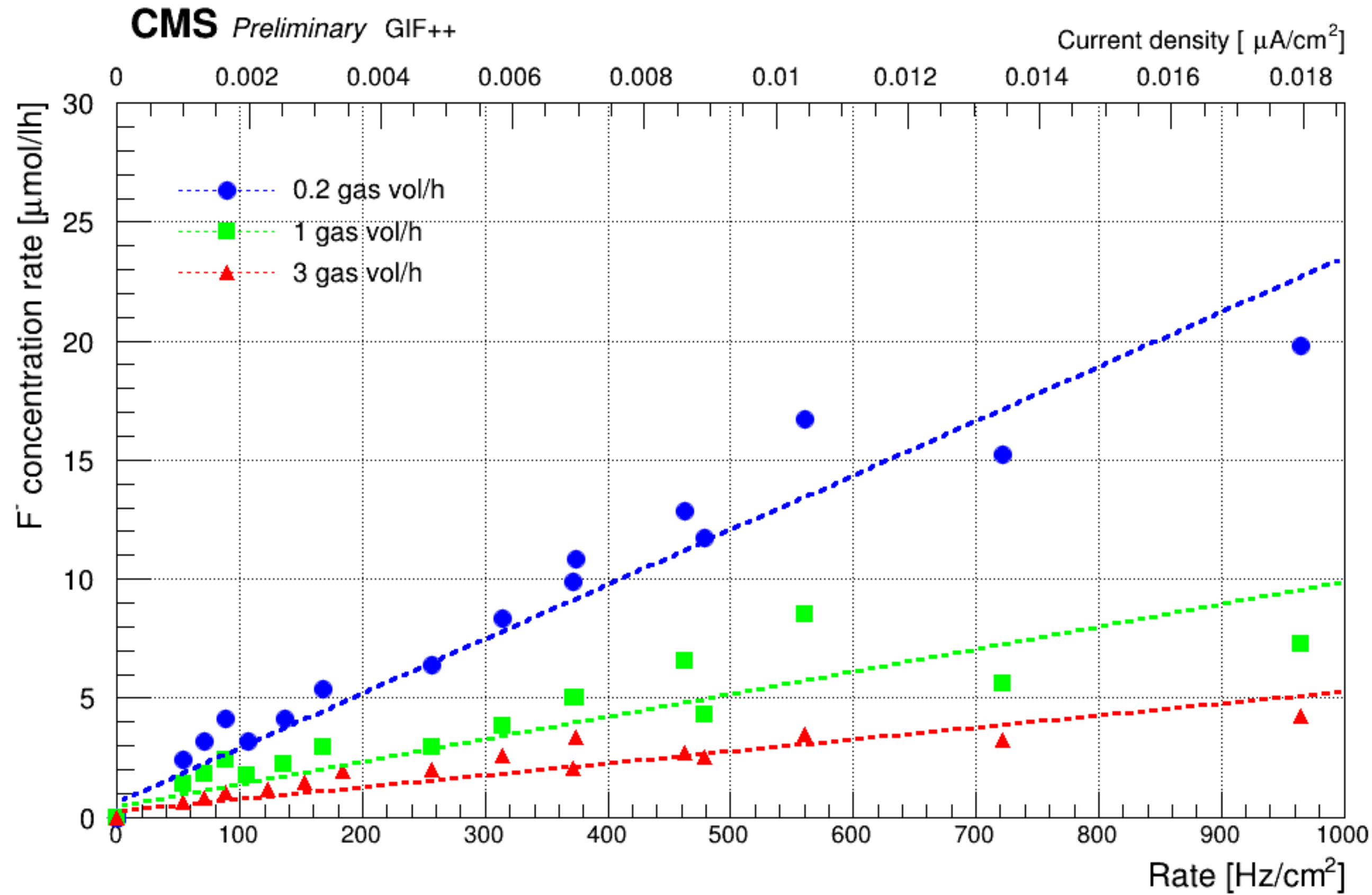
- *HF* high chemical reactivity
- HPL surface damaging by polymerized oil layer
- Surface resistivity decreasing by *HF* deposition, creating a thin conductive layer

HF concentration measured by **ion-selective electrode (ISE)** using **TISAB (Total Ionic Strength Adjustment Buffer)** and distilled water solution in the exhaust gas line

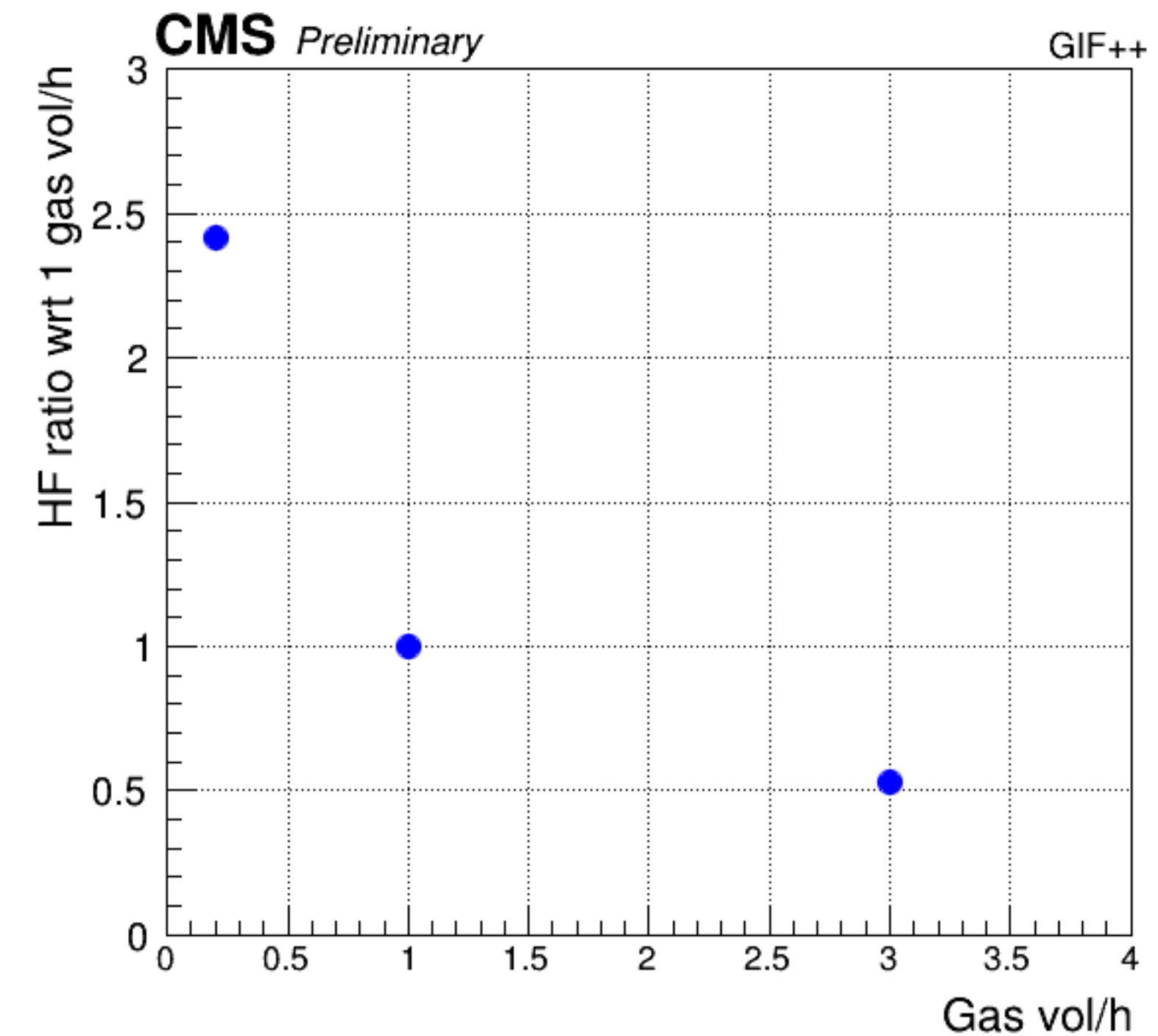


RPC operated at ≈ 9.8 kV and
gas flow fraction analysed 0.5 l/h

HF production @ different background conditions

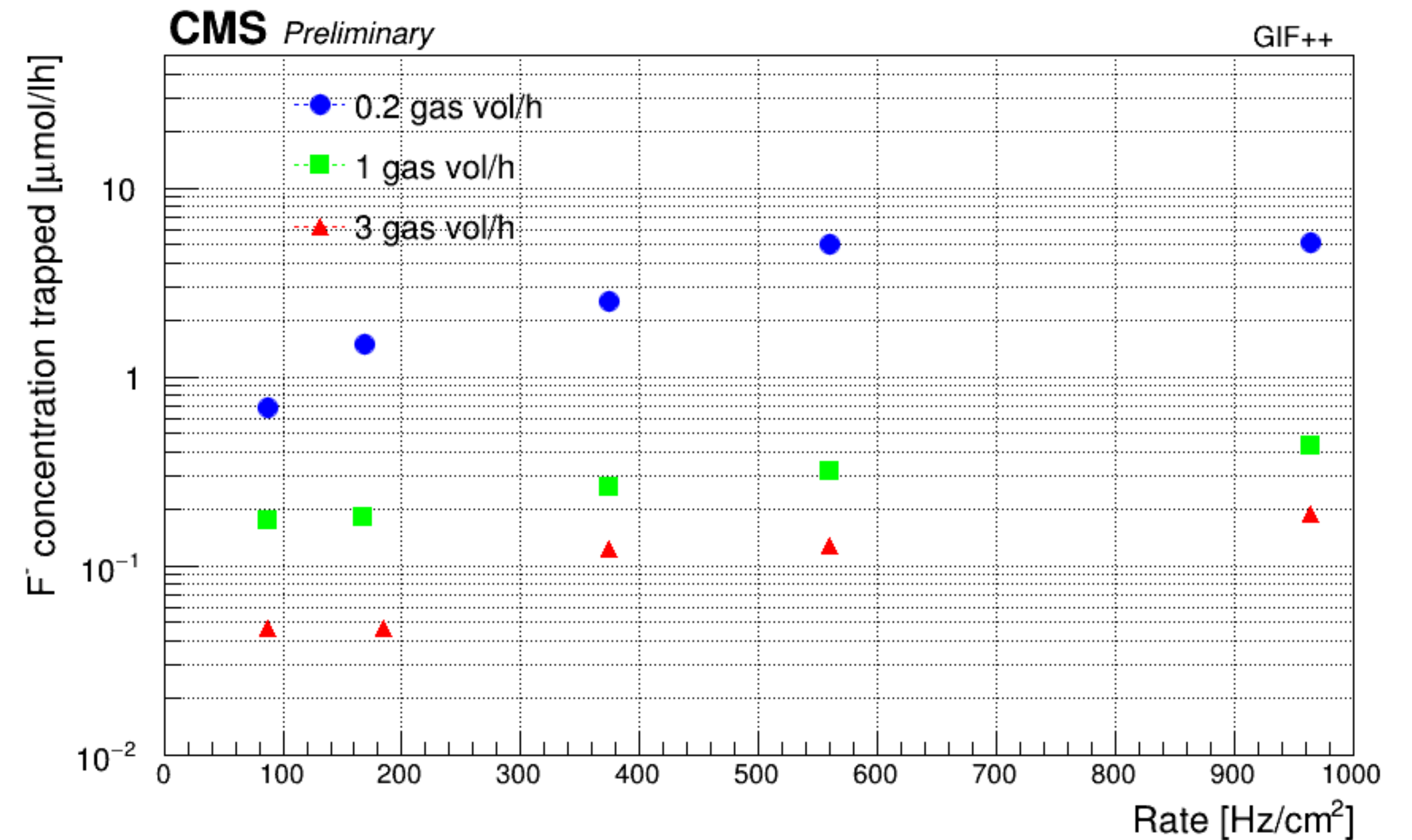


- Linear dependence with the rate (current)
- At higher flow, less HF concentration (different slope for each gas flow applied)



HF trapped @ different background conditions

- HF not efficiently removed by the gas flow and thus trapped inside the gas gap
- HF accumulated during the 8 hours of measurements
- Strong dependence with the high rate (high HF production rates) and gas flow

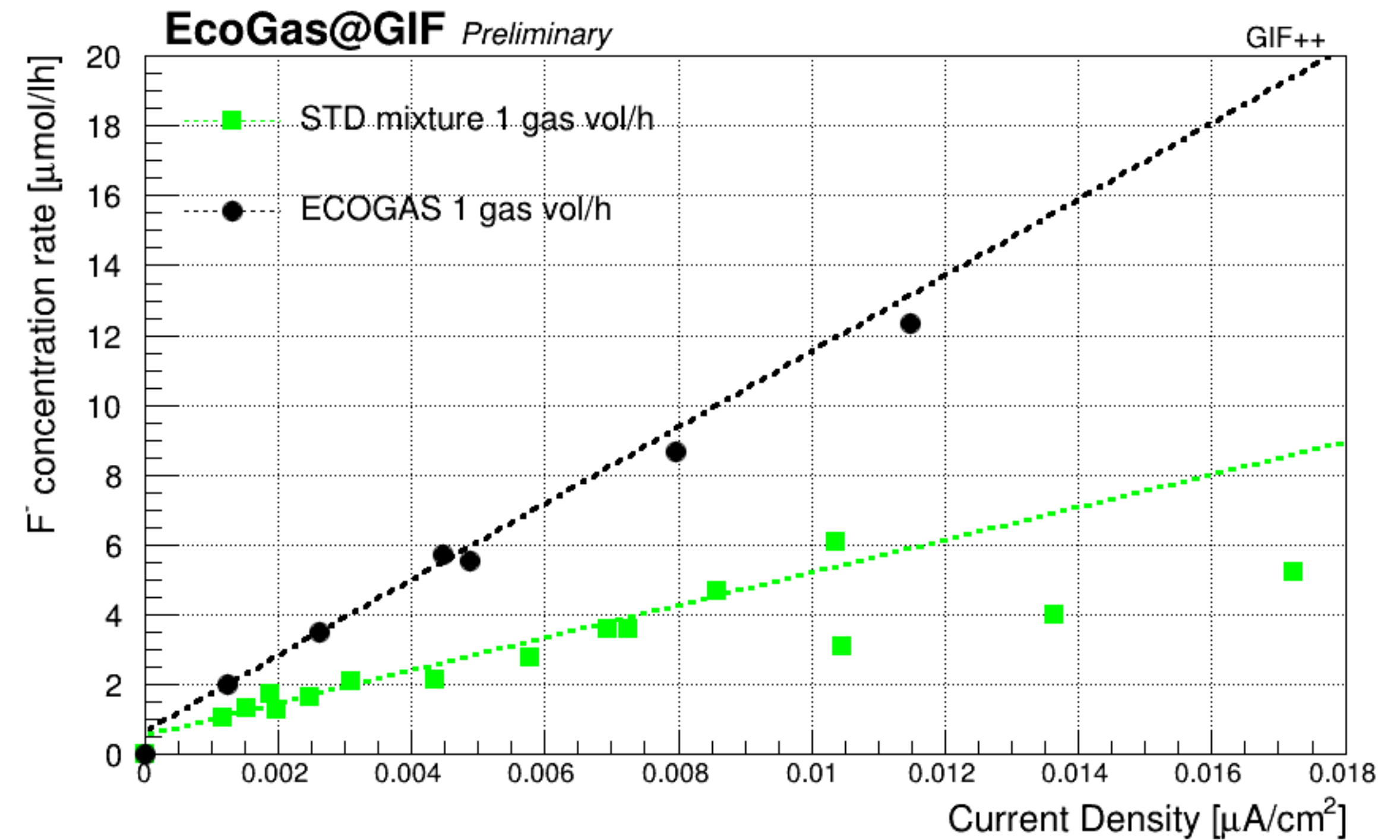


Fluoride concentration rate HFO-based mix

STD: 95.2% C₂H₂F₄, 4.5% iC₄H₁₀ and 0.3% SF₆

ECOgas: 45% HFO-1234ze, 50% CO₂, 4% iC₄H₁₀ and 1% SF₆.

- HF concentration using the ecogas mixture is around 2.5 times greater than the standard
- HFO breaks five times more easily than C₂H₂F₄



CONCLUSIONS

EDS & SEM

- HF deposit on the IRRADIATED samples.
- Detection of Nitrogen could indicate that the linseed oil layer has been partially etched and the melamine is exposed.
- Small amount of Nitrogen could suggest thicker oil layer SOI-3 (white matt) with respect to SOI-2 (IRR).
 - The defect areas (SOI-3) the superficial bakelite layer has been completely etched. In fact, the absence of Nitrogen indicates that the bakelite resin is no longer melamine but phenolic, which is used only for the bulk part.
- Rough topography in IRR samples (halo/mat stein) which could affect the electric field homogeneity.

HF analysis

- HF production dependence with background radiation rate.
- Internal deposition of HF not successfully removed at low gas fluxes.
- HFO molecule breaks more easily than C₂H₂F₄

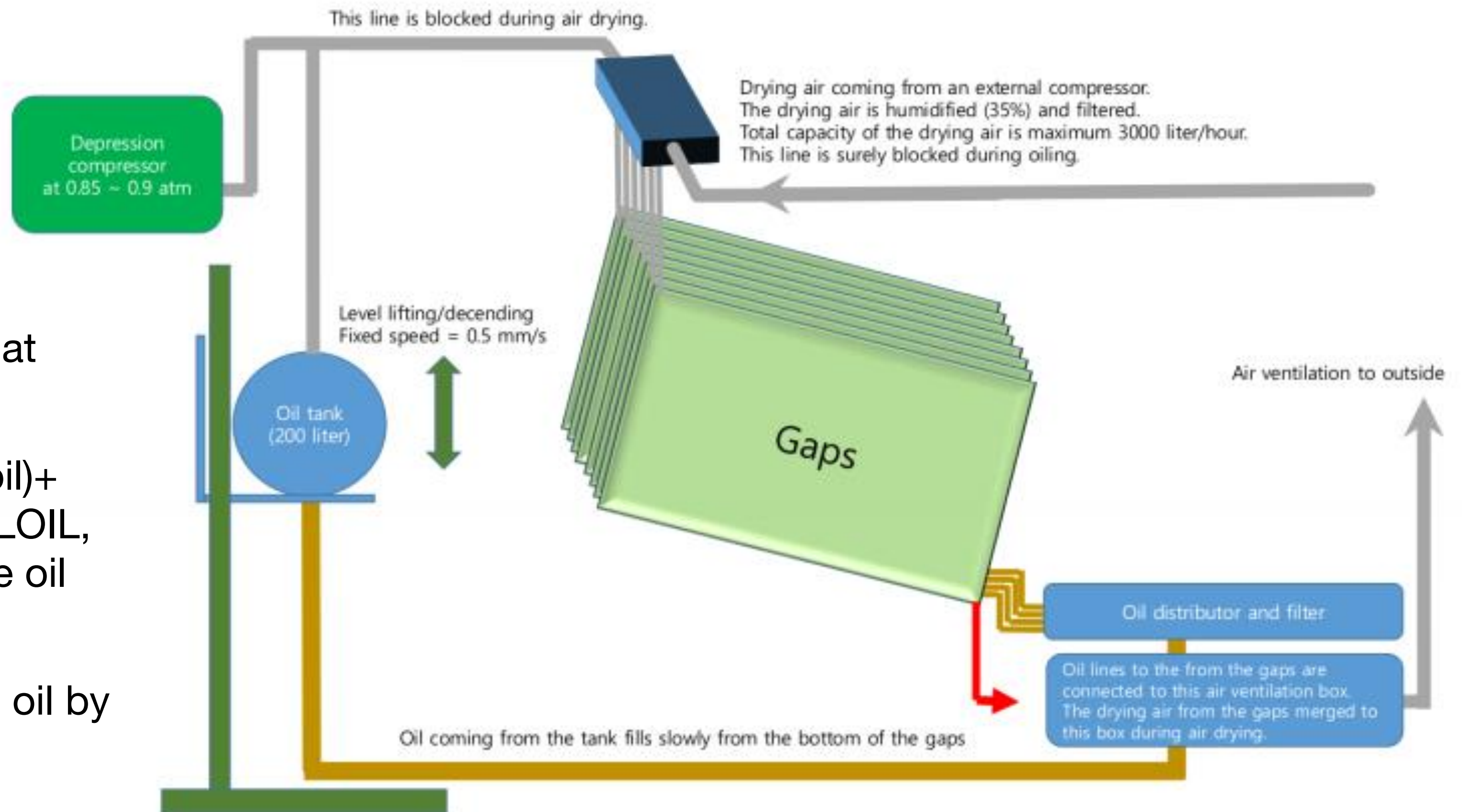
Thanks!

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Backup slides

Gaps production

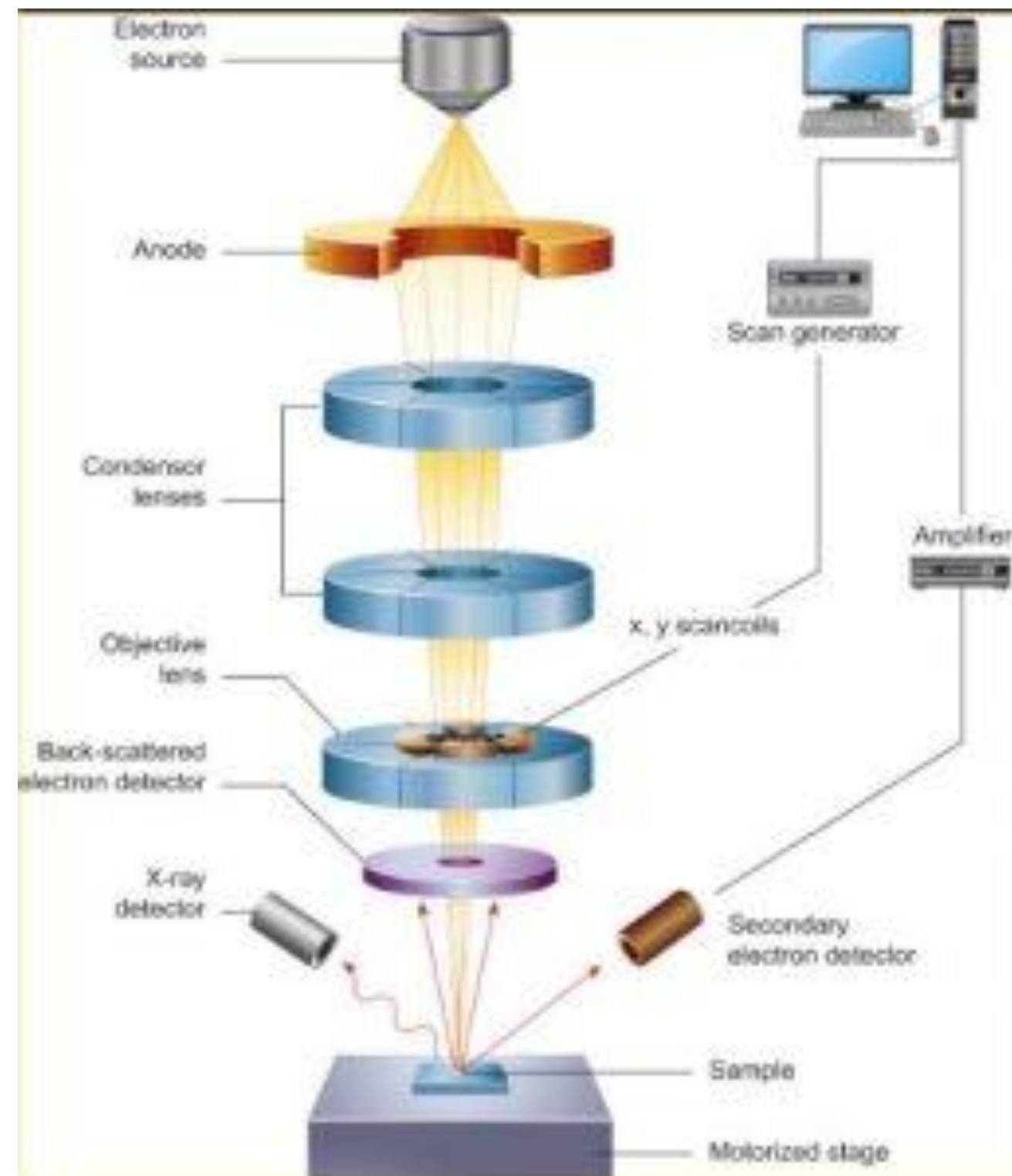
- Spacer material: polycarbonate.
- Glue epoxy 3M DP460.
- Glue dry time (before oiling process): 1-2 days at ambient temperature 20-23 C.
- oil characteristics: 40% oil (drying and boiled oil)+ 60% heptane. The oil for all production is GALLOIL, but different purchase. For 2017 production the oil was the one bought in 2010.
- oiling: 3 hours oiling, where gaps are filled with oil by gravity. Waiting 1 hour for draining out the oil.
- oil drying: 96 hours drying with air T 30-40 C, air humidity 35-40%, with an air flow of 100l/h. The air flow is in the opposite way with respect to the oil flow.



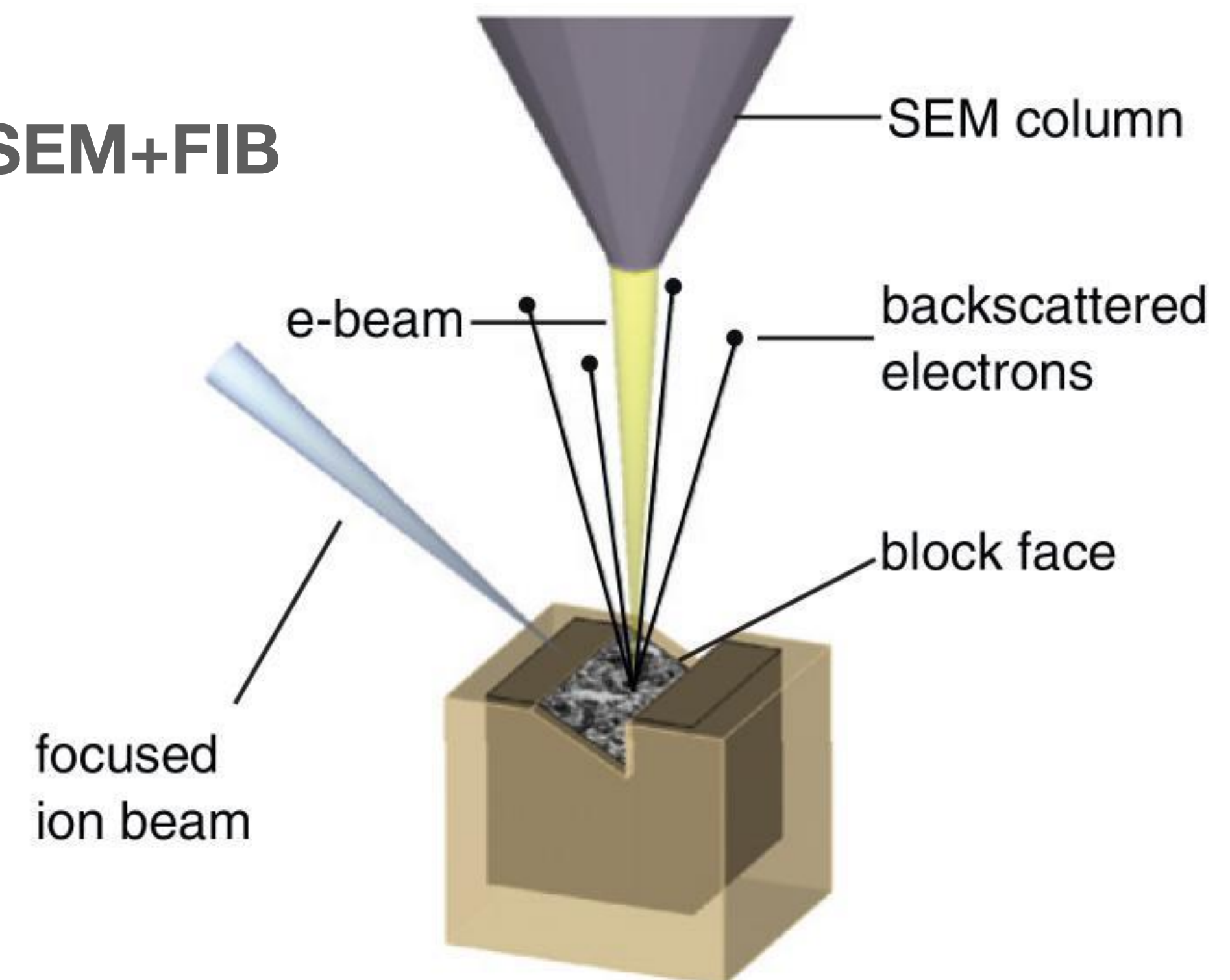
SEM (Scanning Electron Microscope) & FIB (Focused Ion beam)

- **SEM**: surface visual check
- FIB+SEM: superficial and internal surface check
- **FIB**+EDS to check internal bakelite composition

SEM



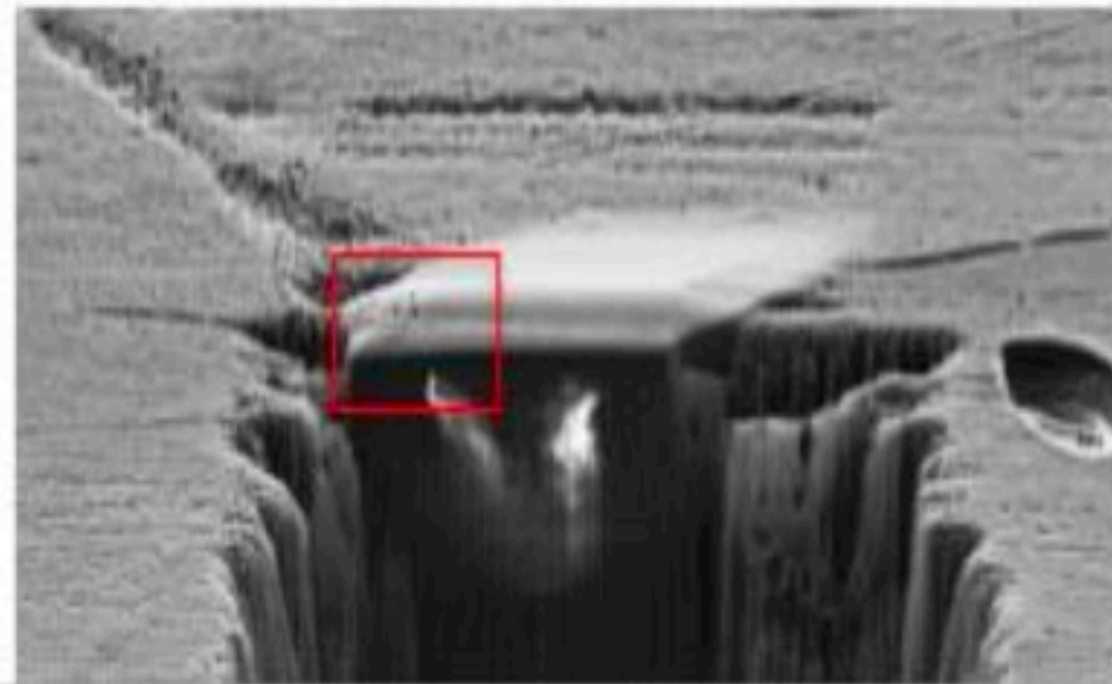
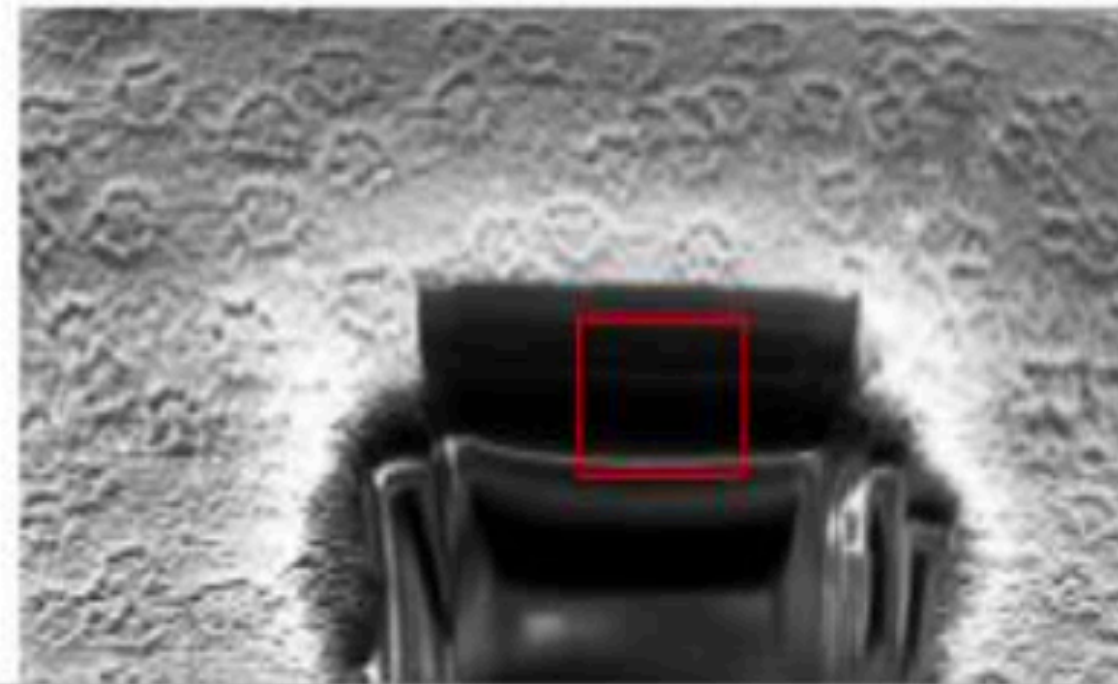
SEM+FIB



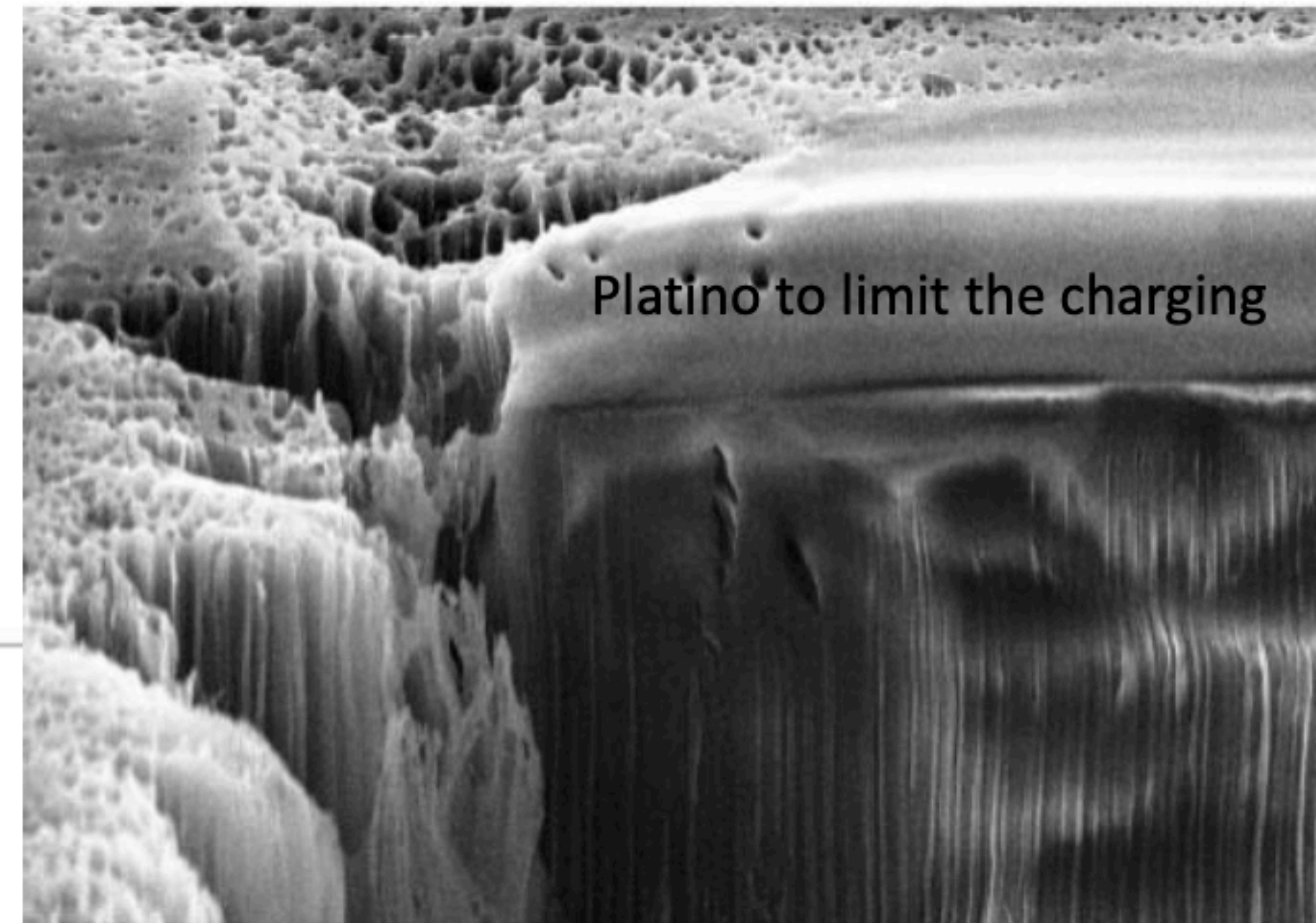
FIB (Focused Ion beam)

Test to verify the **inner bakelite composition and aspect**

- FIB used to remove material layers
- SEM for visual inspection
- EDS to check the chemical composition



FIB+ SEM



Due to the charging problem, the FIB was not stable and the hole not precise. Moreover SEM has been affected.

No evidence of any layer presence

FIB+EDS @ 10um below the surface (SOI-2 &SOI-3): same chemical composition as for the surface.

HF production

