VACUUM EFFECT ON BULK ETCH RATE OF POLYMER TRACK DETECTORS

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EXPERIMENTAL SETUP

- Vacuum chamber of ~30 liters volume (preliminary and turbomolecular pump);
- Target holder - support for PADC detectors (provided by TASL);
- Cf-252 source (20kBq) mounted on the inside part of the window;
- TASL 2 cm x 2.5 cm PADC mounted in the holder.

Fig. 2 Cf-252 sources mounted on the window

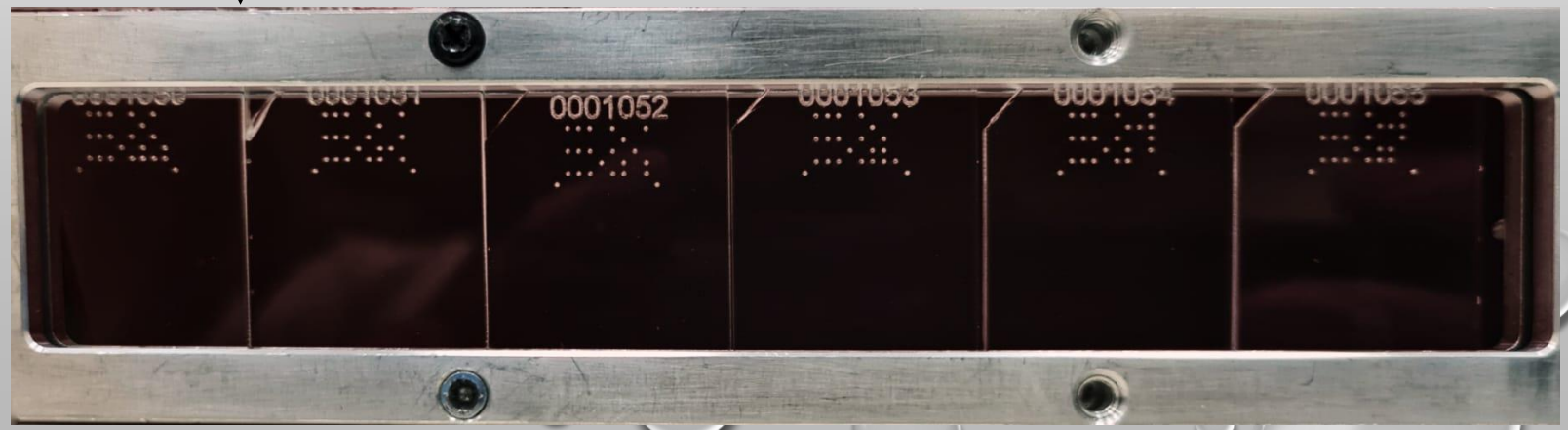
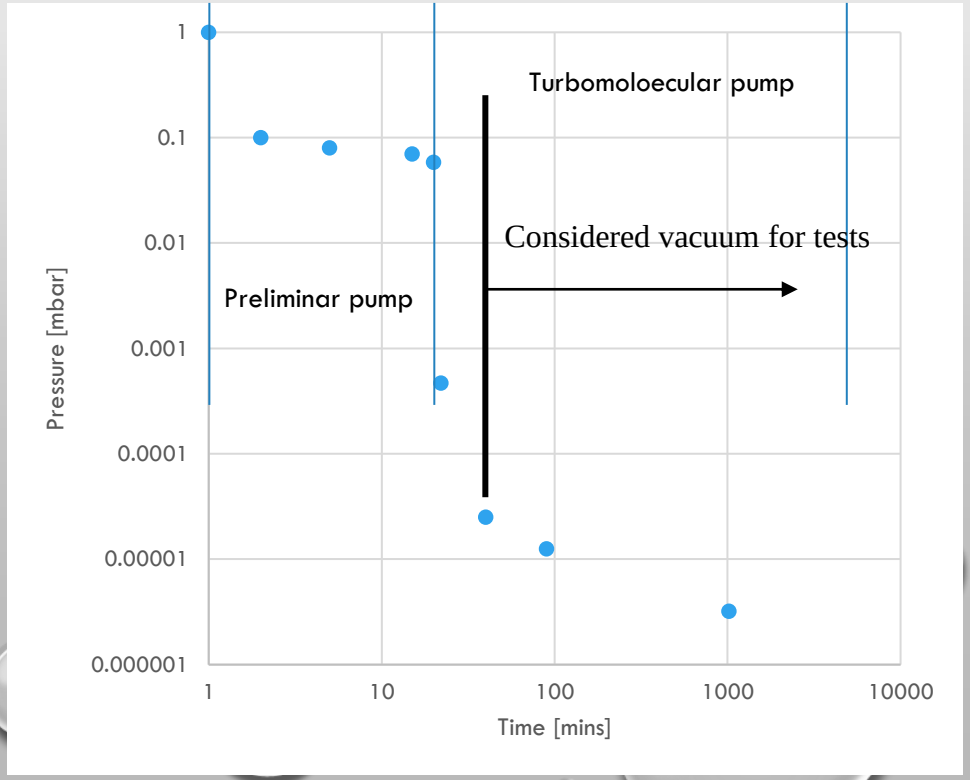


Fig. 3 TASL detectors mounted in the holder

VACUUM EXPOSURE

- 4-6 detectors have been placed in vacuum chamber for each test;
- Preliminary pump achieves the 10^{-2} mbar range in 20', and the turbomolecular one goes to 10^{-5} mbar in 2';
- Vacuum times varied from 1 minute to almost 17 hours;
- There should be no changes induced by the vacuum conditions before irradiation is taking place [3];
- For each test we've been working in two cases: with or without Cf-252 source (for fission fragments or mass difference assessment methods);
- When the vacuum pressure achieved 10^{-5} mbar, the detectors have been places down for uniform irradiation;

Fig.4 Approximation of vacuum pressure trend



VACUUM EFFECT ON DETECTOR'S MASS

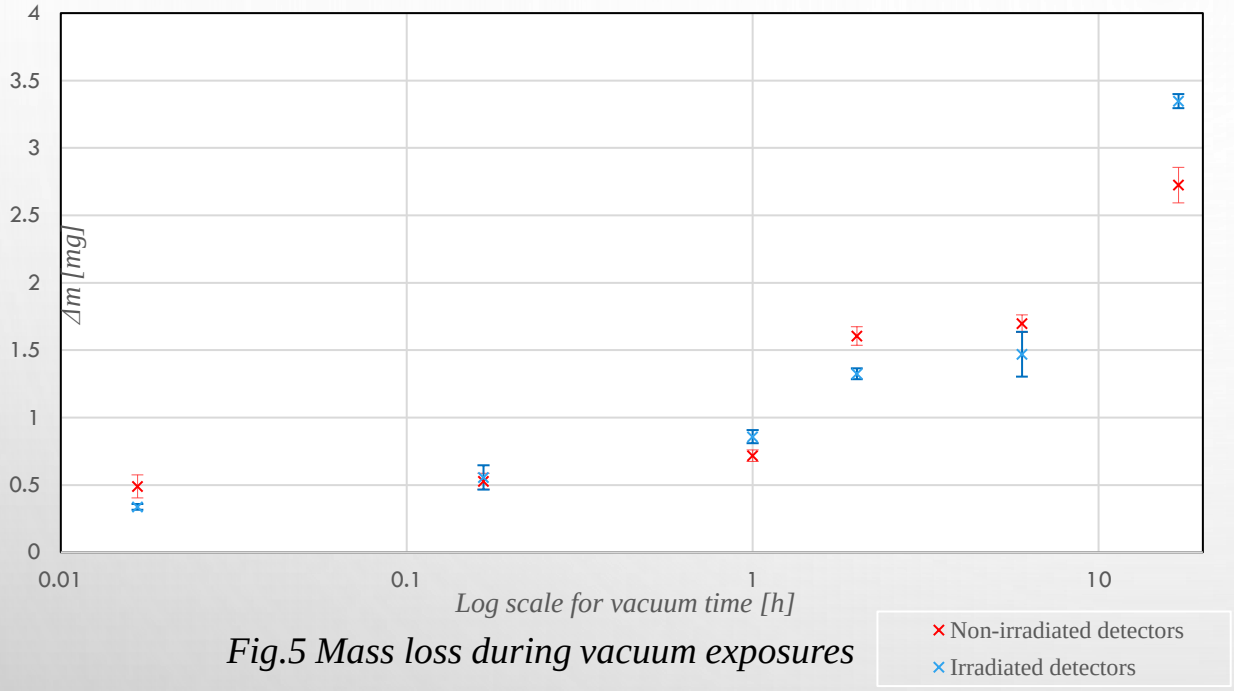


Fig.5 Mass loss during vacuum exposures

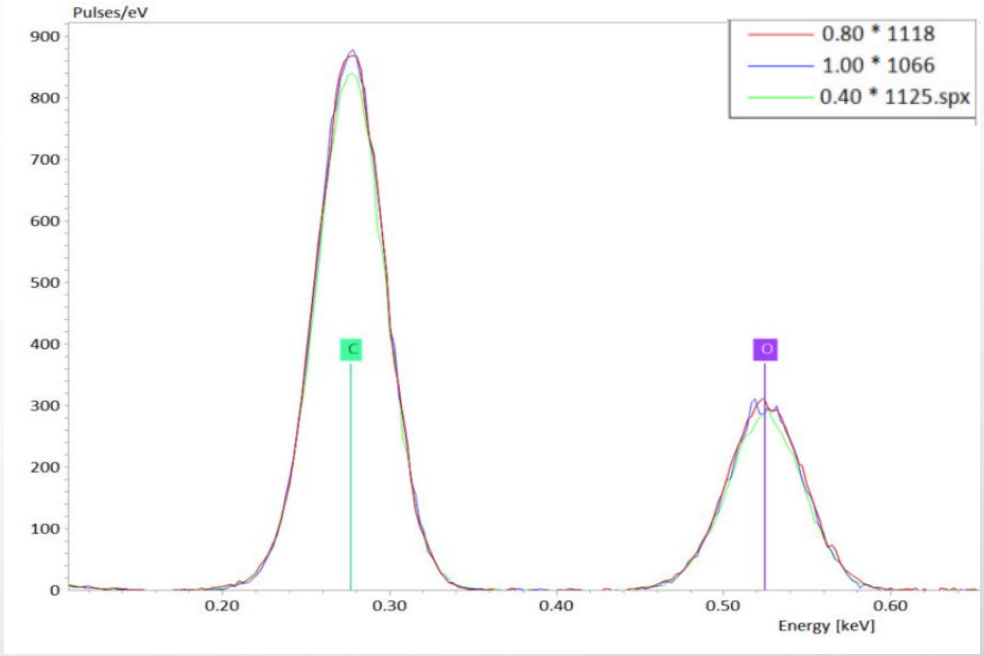


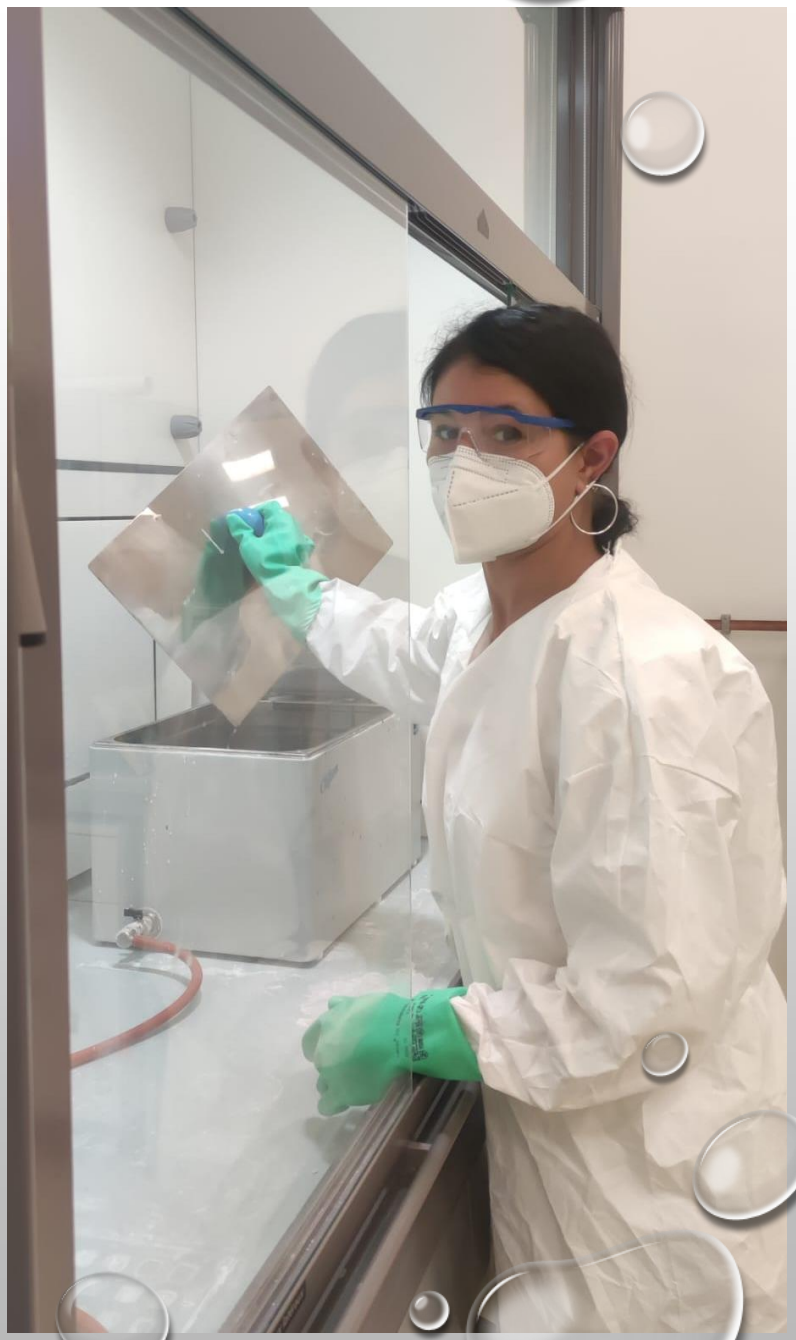
Fig.6 SEM analysis for vacuum exposed detectors

Sample code	Context	Atomic concentrations	
1066	Refference sample – kept in the frigde	69.83 ± 1.71	30.17 ± 1.08
1118	No irradiation performed, 6h in vacuum	69.50 ± 1.70	30.50 ± 1.09
1125	Irradiated, 6h in vacuum	70.24 ± 1.07	29.76 ± 1.72

- Performing aditional irradiation in vacuum did not enhance the material loss process;
- Mass loss could be explained by Hidrogen's desorption or of both C and O in the same ratio.
- The samples have been measured after the vacuum exposure and 20 hours later --► no increase in mass (no re-absorption effect has been induced post-vacuum exposure)

ETCHING PROCEDURE

- 6.25 M NaOH solution (20 litres), 85^{o}C;
- Assumptions from previous tests: bulk etch rate for TASL detectors (PADC of 2 cm x 2.5 cm bevel-cut forms) – V_b ~ 5 µm/h;
- Every etching step included control detectors to estimate the bulk etch rate during each chemical processing;
- For the irradiated detectors we've tried not to remove more than 10 µm in order to apply the fission fragment technique [1];



Bulk etch rates for non-irradiated detectors

No Irradiation performed – V_b through mass difference method			
Time	*V_b [µm / h]*	**Control V_b [µm / h]*	*Relative difference*
1 min	4.068 ± 0.056	4.351 ± 0.116	- 6.5 %
10 min	3.926 ± 0.014	4.305 ± 0.068	- 8.8 %
1 h	4.802 ± 0.185	4.973	- 3.44 %
2 h	4.676 ± 0.164	4.351 ± 0.116	7.47 %
6 h	3.779 ± 0.089	4.351 ± 0.116	- 2.4 %
~17 h	4.382 ± 0.129	4.973	- 11.88 %

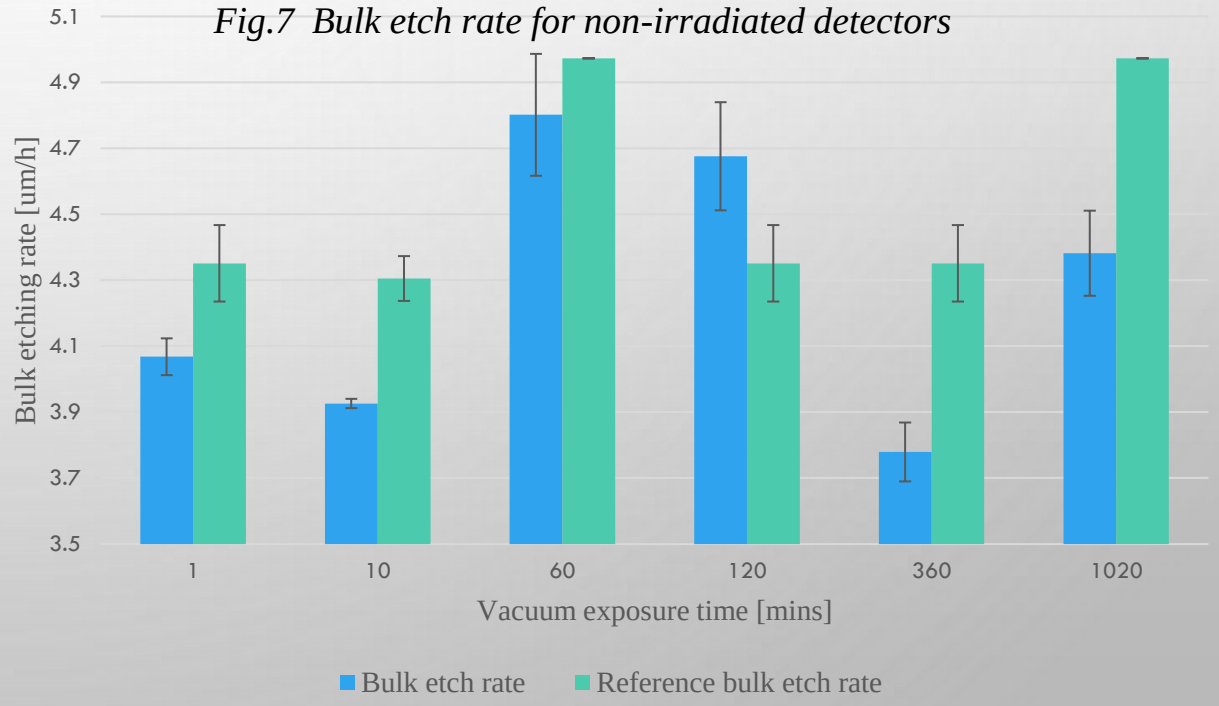
** Control bulk etch rate assessed through mass difference method*

Assessment through mass difference method:

$$V_B = \frac{m_{after\ vacuum} - m_{after\ etch}}{2A\rho t} \quad [1]$$

- constant density (ρ) of 1.31 g/cm^3;
- constant area (A) of 477 mm^2;
- t = 2 h of etching;

Fig.7 Bulk etch rate for non-irradiated detectors



Bulk etch rates for irradiated detectors

Irradition performed with 20 kBq Cf-252 source - V_b through fission fragments method			
Time	*V_b [µm / h]*	**Control V_b [µm / h]*	*Relative difference [%]*
1 min	4.729 ± 0.037	4.305 ± 0.068	9.85 %
10 min	4.119 ± 0.020	4.351 ± 0.116	- 5.33 %
1 h	4.521 ± 0.117	4.973	- 9.09 %
2 h	4.107 ± 0.011	4.351 ± 0.116	- 5.61 %
6 h	4.11 ± 0.029	4.351 ± 0.116	- 5.54 %
~17 h	4.052 ± 0.025	4.351 ± 0.116	- 6.87 %

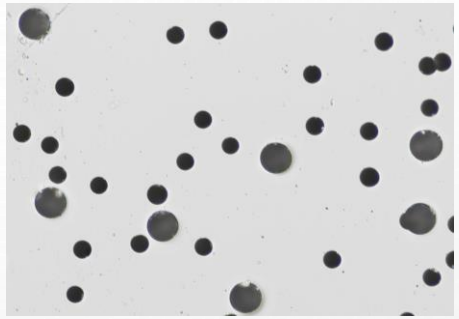
** Control bulk etch rate assessed through mass difference method*

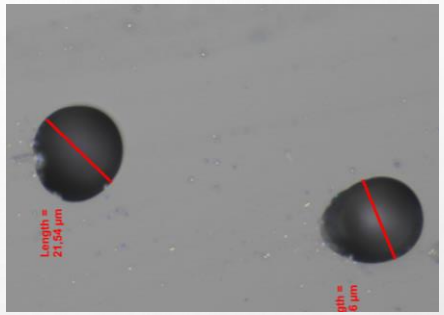
$$V = \sqrt{1 + \left(\frac{D}{2h}\right)^2 \left(\frac{2}{1 - \left(\frac{d}{2h}\right)^2}\right)^2}$$ [2]

→ ***V = 1.195 ± 0.052***

for a removed layer h = 8.22 µm

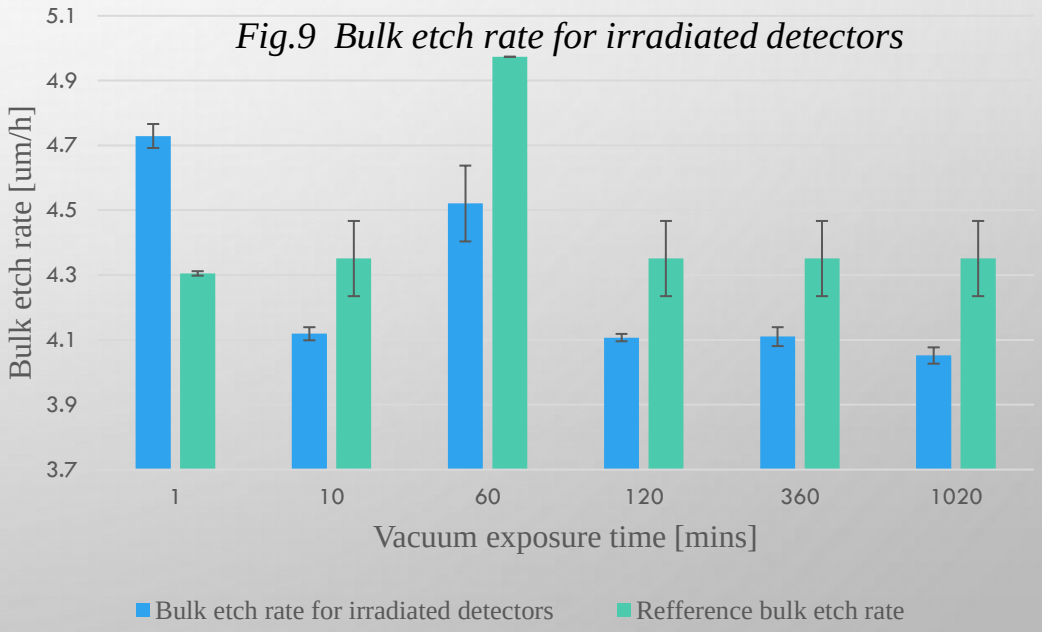
Fig. 8 Alpha particles and fission fragments tracks





$$D_{ff} = 2 * Vb * t$$ [2]

Fig.9 Bulk etch rate for irradiated detectors



SEM analysis of etched detectors

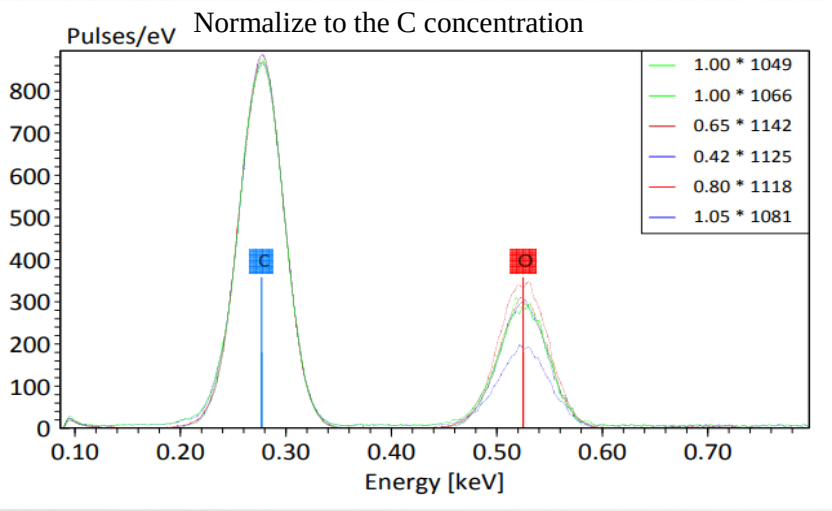


Fig.10 C&O spectra throughout SEM assessment

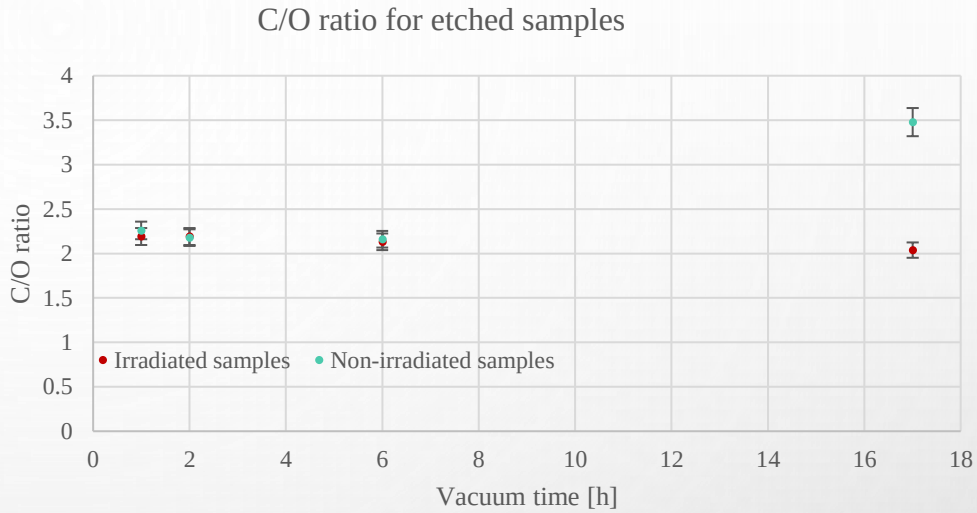


Fig.11 C/O atomic ratios for the etched detectors

Sample code	Context	Atomic concentrations	
1066	Reference sample – kept in the frigde	69.83 ± 1.71	30.17 ± 1.08
1118	No irradiation performed, 6h in vacuum	69.50 ± 1.70	30.50 ± 1.09
1081	No irradiation performed, 17h in vacuum, etched	77.67 ± 1.93	22.33 ± 0.85
1066	Reference sample etched	69.83 ± 1.71	30.17 ± 1.08
1125	Irradiated, 6h in vacuum, etched	70.24 ± 1.07	29.76 ± 1.72
1142	Irradiated, 17h in vacuum, etched	67.09 ± 1.63	32.91 ± 1.16

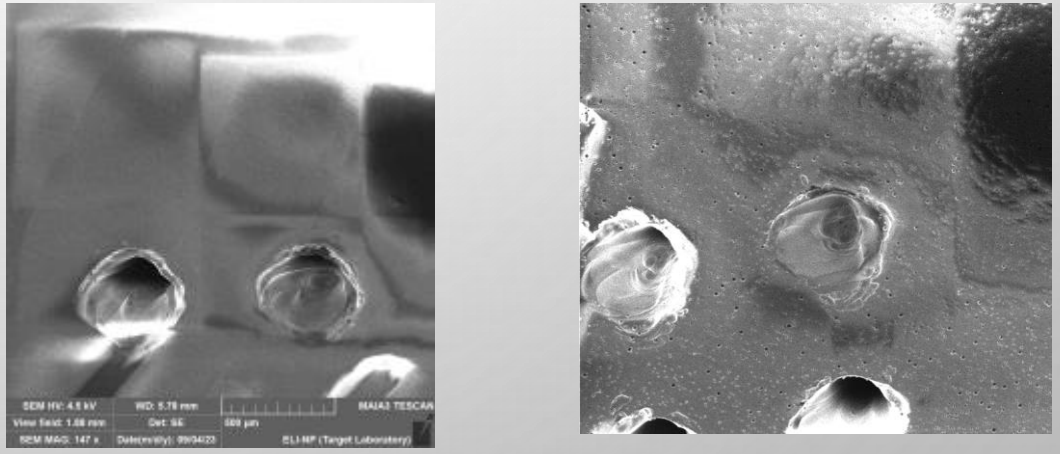


Fig.12 Alpha particles tracks from SEM analysis

CONCLUSION & OUTLOOK FOR FUTURE RESEARCH

1) Mass loss increases with the time of vacuum exposure (and pressure), which may affect the assumption used in mass difference method. → *Constant vacuum pressure-time steps should be considered, in order to establish a correlation between the pressure level, exposure time and mass loss.*
2) There is no a so called "elastic effect" induced by the vacuum – detectors prove the same lower mass for hours later after the extraction. → *Look for parameters that may induce temporary stress in the material.*
3) Bulk etch rate changes have been noted when irradiation is performed in vacuum. Most of the cases (w/ 2 exemptions) have shown lower values for detectors bulk etch rate comparing to the control ones. → *As long as there is no re-absorption effect, all the detectors should be etched together.*
4) No explicit impact of additional irradiation has been proven on the mass loss process during vacuum time comparing to the case when no irradiation has been performed.
5) Samples simply kept in vacuum have shown an increasing C/O ratio (+46%) after the etching, while the vacuum + irradiated ones proved a lower value for the same ratio (-15%); → *Further studies need to be performed for sensitivity assessment / activation energy? -> EURADOS CR-39 Quality Task Studies?*

BIOGRAPHY

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Thank you for your attention!