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REFERENCE
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Metallurgy

Microscope inspection of oiled High Pressure Laminated (HPL) Bakelite

Summary:

RPC (Resistive Plate Chamber) detector was irradiated at GIF++ with gammas. After having collected around 20 mC/cm2 of integrated charge, two "gaps" over three, started to give high dark currents. The panels are up to 2m long and present a layer of "Bakelite" (2 mm) made by Kraft paper impregnated with melamine/phenol resins. Internal electrode surface covered with a thin linseed oil layer (~µm).

When the gaps have been opened some matt spots around the spacers were visible, and in addition, in the two problematic gaps there are big matt spots on the edge. A non-irradiated gap has been opened to use as a reference. No any matt spots have been observed, even if it is possible to notice a halo around the spacers.

It is aimed that the insights of the microscope inspection are helping to determine the origin of the colour contrast at some specific regions.

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1. Introduction

RPC (Resistive Plate Chamber) detector was irradiated at GIF++ with gammas. After having collected around 20 mC/cm2 of integrated charge, two "gaps" over three, started to give high dark currents. The panels are up to 2m long and present a layer of "Bakelite" (2 mm) made by Kraft paper impregnated with melamine/phenol resins. Internal electrode surface covered with a thin linseed oil layer (~µm).

1.1 Aim of the study

When the gaps have been opened some matt spots around the spacers were visible, and in addition, in the two problematic gaps there are big matt spots on the edge (Figure 1).

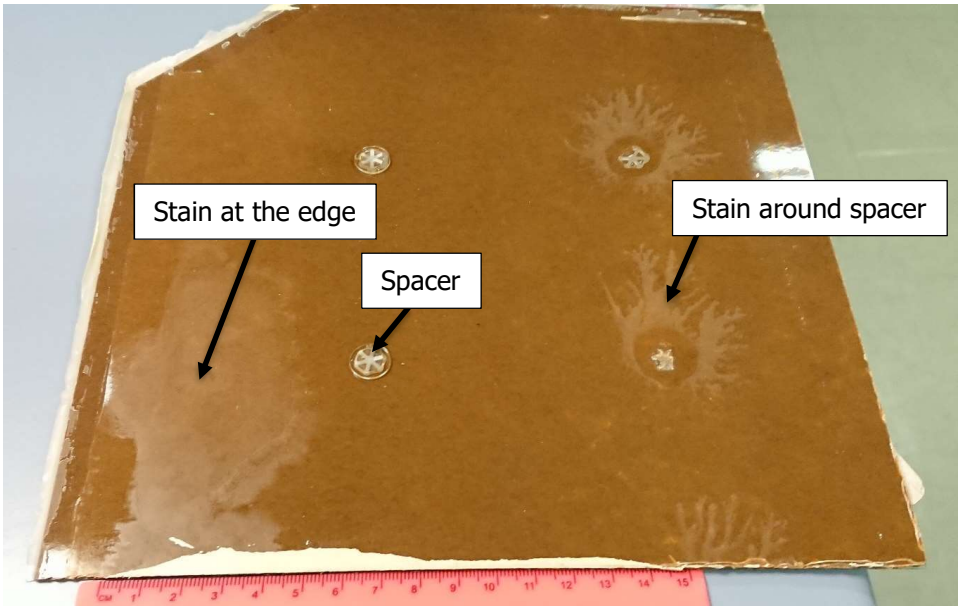


Figure 1 – Visual inspection of sample with matt spots around the spacers and at the edge area

A non-irradiated gap has been opened to use as a reference. No any matt spots have been observed, even if it is possible to notice a halo around the spacers.

It is aimed that the insights of the microscope inspection are helping to determine the origin of the colour contrast at some specific regions.

1.2 Key words

SEM, EDS, FIB, Bakelite

2. Protocol

2.1 Samples

The original panels are up to 2m long and present a layer of "Bakelite" (2 mm) made by Kraft paper impregnated with melamine/phenol resins. Internal electrode surface covered with a thin linseed oil layer (~µm). The panels were cut in order to have manageable dimensions (approximately 30 mm x 30 mm). The cut was performed in dry with a guillotine in order to avoid external pollution, and anyway, the samples were covered with protective foils to reduce features due to the cutting but also handling and/or transport.

In the first instance, reference sample (identified as Sample #1) and two samples from TW LG1.4 (identified as Sample #2 and Sample #3). Based on the findings, a new set of samples (Samples #4 to Sample #9) was prepared in the same way and inspected by means of microscopy techniques.

A maximum of four sites of interest (SOI) were defined in each sample. Representative example is shown in Figure 2 (but not all samples presented the four SOI described).

SOI-1	Spacer glue area
SOI-2	Area around spacer
SOI-3	Halo/Matt stain
SOI-4	Non-affected area

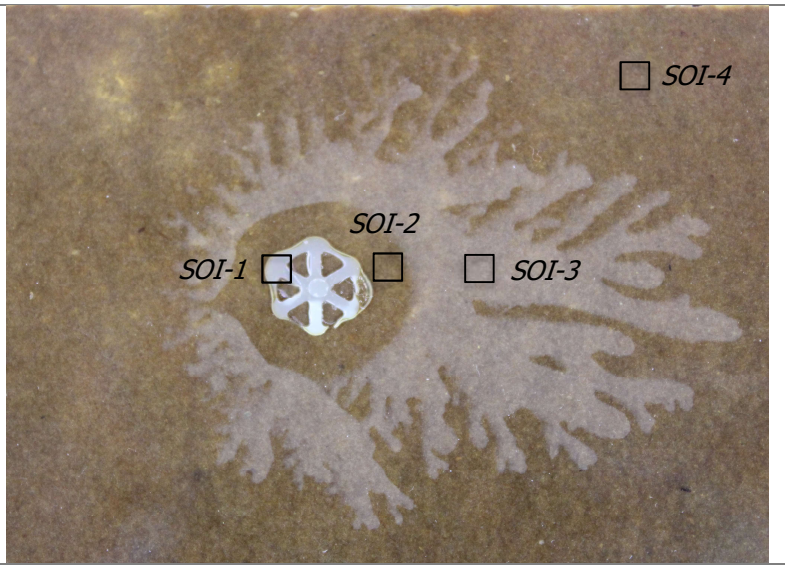


Figure 2 — Example of the SOI locations around

The identification and details of the studied samples is included in Table 1.

Table 1 – Samples information

Sample ID	CODE	Gap Name	Description	A/C
Sample #1	1.4 REF 2	KODEL 1.4 BOT LEAK	Produced in 2017. Never irradiated	Anode
Sample #2	TW LG1.4_2	KODEL 1.4 TW	Produced in 2017. Irradiated (with big stain)	Anode
Sample #3	TW LG1.4_1	KODEL 1.4 TW	Produced in 2017. Irradiated (with big stain)	Anode
Sample #4	RE1/2 No oil	RE1/2 P517PV code 128B	RE1/2 No oil	-
Sample #5	TN LG1.4 1	KODEL 1.4 TN	Produced in 2017. Irradiated, with a spot around the marker, but working properly	Anode
Sample #6	TN LG1.4 2	KODEL 1.4 TN	Produced in 2017. Irradiated, with a spot around the marker, but working properly	Cathode
Sample #7	RE4/2 IRR5	KODEL_CMS_RE4_2_TN116	Produced in 2010. Irradiated at GIF (40 mC/cm2)	Anode
Sample #8	RE4/2 REF3	KODEL_CMS_RE4_2_TN110	Produced in 2010. Never irradiated	Anode
Sample #9	RE1/2 REF4	KODEL_CMS_RE1/2_TN23	Produced in 2005. Never irradiated	Anode

2.2 Equipment

- Scanning Electron Microscope (SEM), field emission gun FEG Sigma (ZEISS) with InLens (Secondary Electron), Evan-Thornley Secondary Electron (SE2), and back-scattered electron (AsB) detectors for imaging.
- Focused Ion Beam (FIB)/SEM Zeiss XB540 with Secondary Electron Secondary Ion (SESI), Energy Selective Backscattered (ESB) and Back Scattered Detector (BSD) detector.
- 50 mm2 X Max EDS detector (Oxford), AZTEC software. EDS detection: Makes impossible to detect presence of elements below around 0.1-0.5 wt. % (the value depends on the weight and the matrix around), or light elements (impossible below Z= 4 and only large amount for Z between 4 and 11).
- CASINO Monte Carlo electron trajectory simulation software. Simulation was performed to estimate the interaction volume at 20 keV beam energy in bulk carbon. The penetration depth obtained was approximately 1.5 µm.
- The charging made impossible to capture uniform images of the specimens. To solve this problem, a sample metallization with gold or carbon was performed in all samples to increase the conductivity of the surface so that sufficient electrons can escape and avoid charging.

3. Experimental work and results

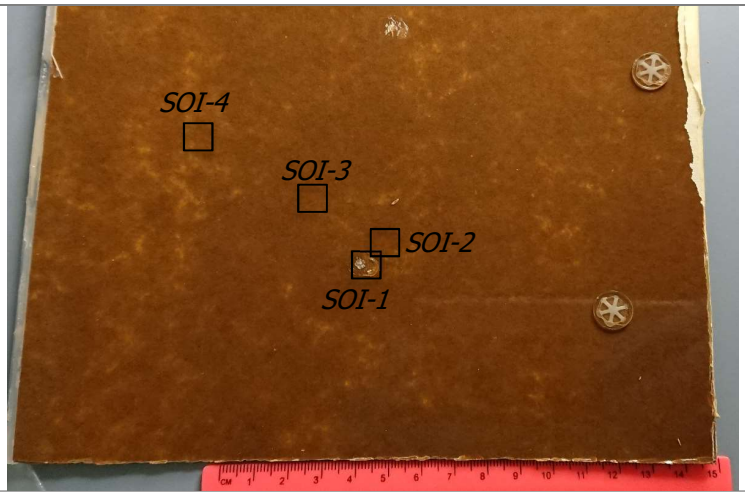
3.1 Microscope inspection on Sample #1, Sample #2 and Sample #3

An exhaustive inspection was performed in Sample #1, Sample #2 and in Sample #3.

Semi-quantitative EDS analysis at 20 keV was performed on their surface in different SOI and the location and results are shown in Figure 4 (Sample #1), Figure 4 (Sample #2) and Figure 5 (Sample #3)). Note that all the percentages correspond to normalized weight percentage (wt. %) of the detected elements carbon (C), nitrogen (N), oxygen (O) and fluorine (F).

- F was only detected on the irradiated samples (Sample #2 and Sample #3), not at the reference (Sample #1);
- The glue composition was determined in SOI-1 in Sample #2 as the remaining volume is thick enough avoiding any influence of the layers below on the EDS spectrum. It is mainly rich in C and O;
- The halo in Sample #1, not irradiated, presents C, N and O while same area after irradiation in Sample #2 presents only C and O;
- Irradiated and non-irradiated present the same composition around the spacer (SOI-2) and the non-affected area (SOI-4).
- The big stain close to the edge in Sample #3 (SOI-2) presents N, C and O.

SOI-1	Spacer glue area
SOI-2	Area around spacer
SOI-3	Halo
SOI-4	Non-affected area



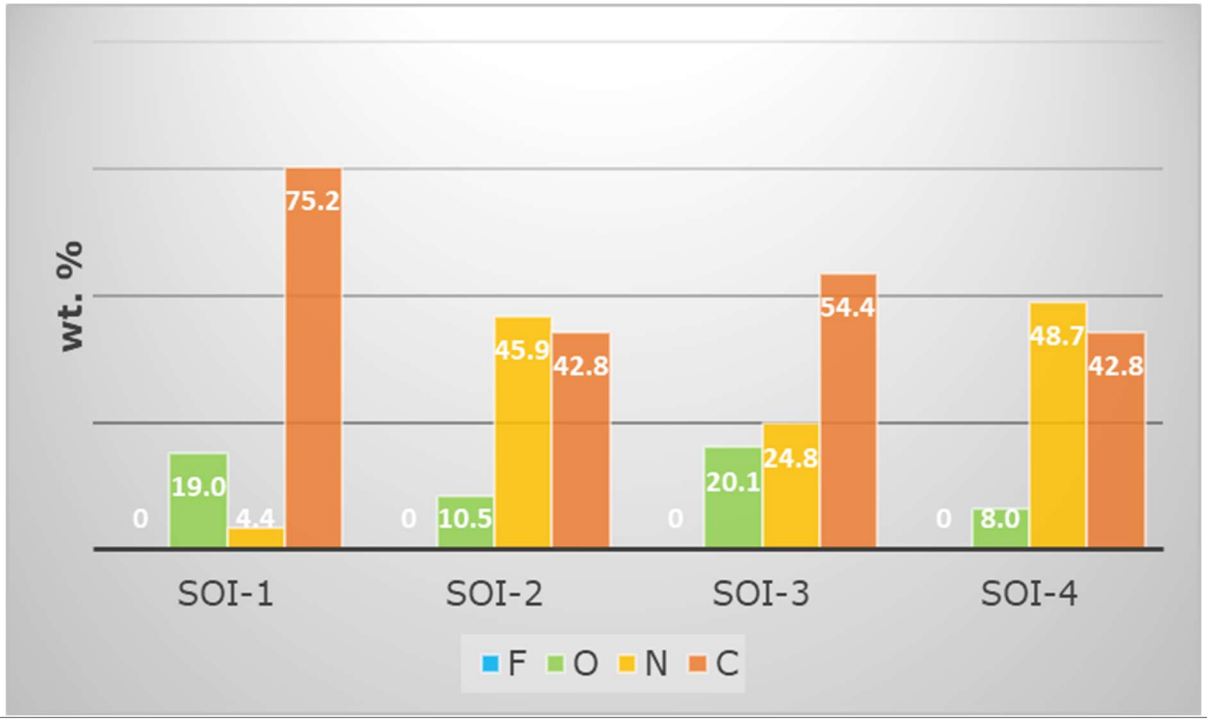
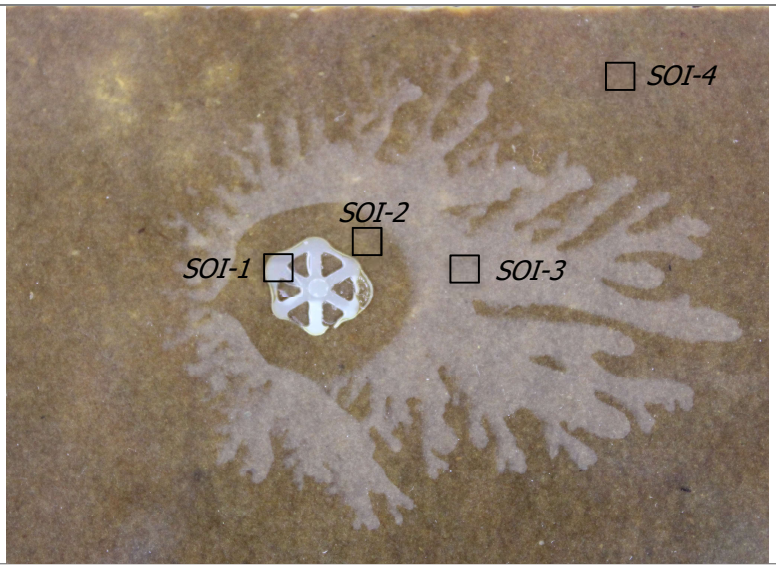


Figure 3 —SOI locations in Sample #1 and EDS results in wt. %

SOI-1	Spacer glue area
SOI-2	Area around spacer
SOI-3	Halo/ Matt stain
SOI-4	Non-affected area



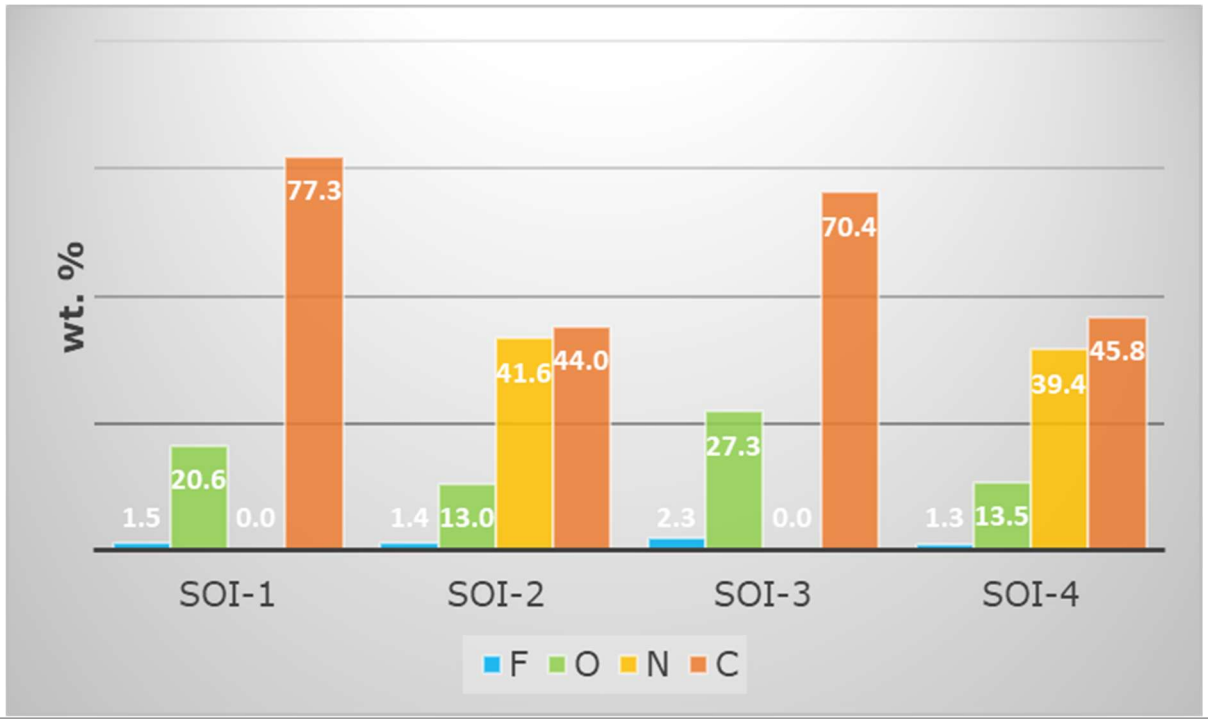
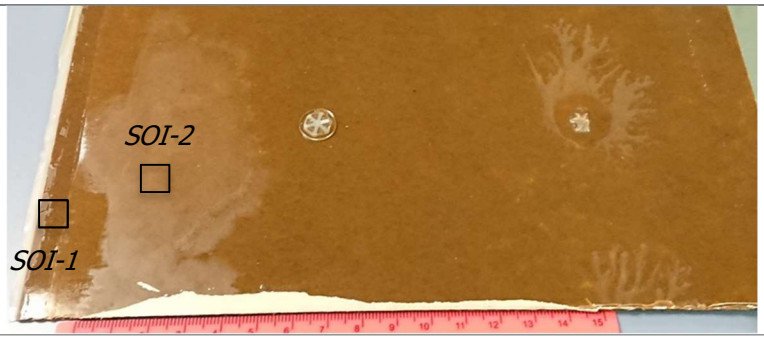


Figure 4 —SOI locations in Sample #2 and EDS results in wt. %

SOI-1	Edge glue area
SOI-2	Halo/ Matt stain



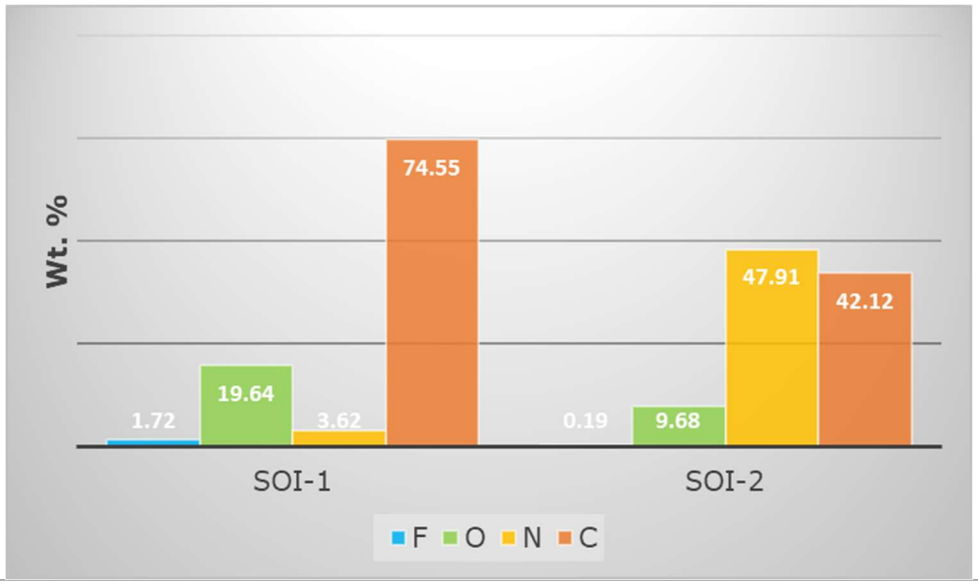


Figure 5 —SOI locations in Sample #3 and EDS results in wt. %

Additionally, the surface characteristics at the non-affected area and at a randomly selected stain was compared by SEM imaging on Sample #2 presenting the first one a smooth aspect while the second one shows a rough topography. Representative images are displayed in **Error! Reference source not found.**.

In an attempt to better understand the origin of the observed stains, cross section was prepared on two locations (SOI-2 and SOI-3) by FIB cross sectional milling initially depositing a carbon protection barrier at a milling current of 300 pA and accelerating voltage of 30 keV. Coarse milling was then performed at a milling current of 15 nA and accelerating voltage of 30 keV. Polishing of the revealed surface was then performed at a current of 3 nA and accelerating voltage of 30 keV. (See Figure 7).

It was noticed that in SOI-3, a superficial layer of approximately 500 nm was visible as it is shown in Figure 8 while it is not appreciable in SOI-2 (Figure 9).

The EDS analysis performed on this layer was not conclusive, detecting C, O and F like in the analyses performed from top view in SOI-3.

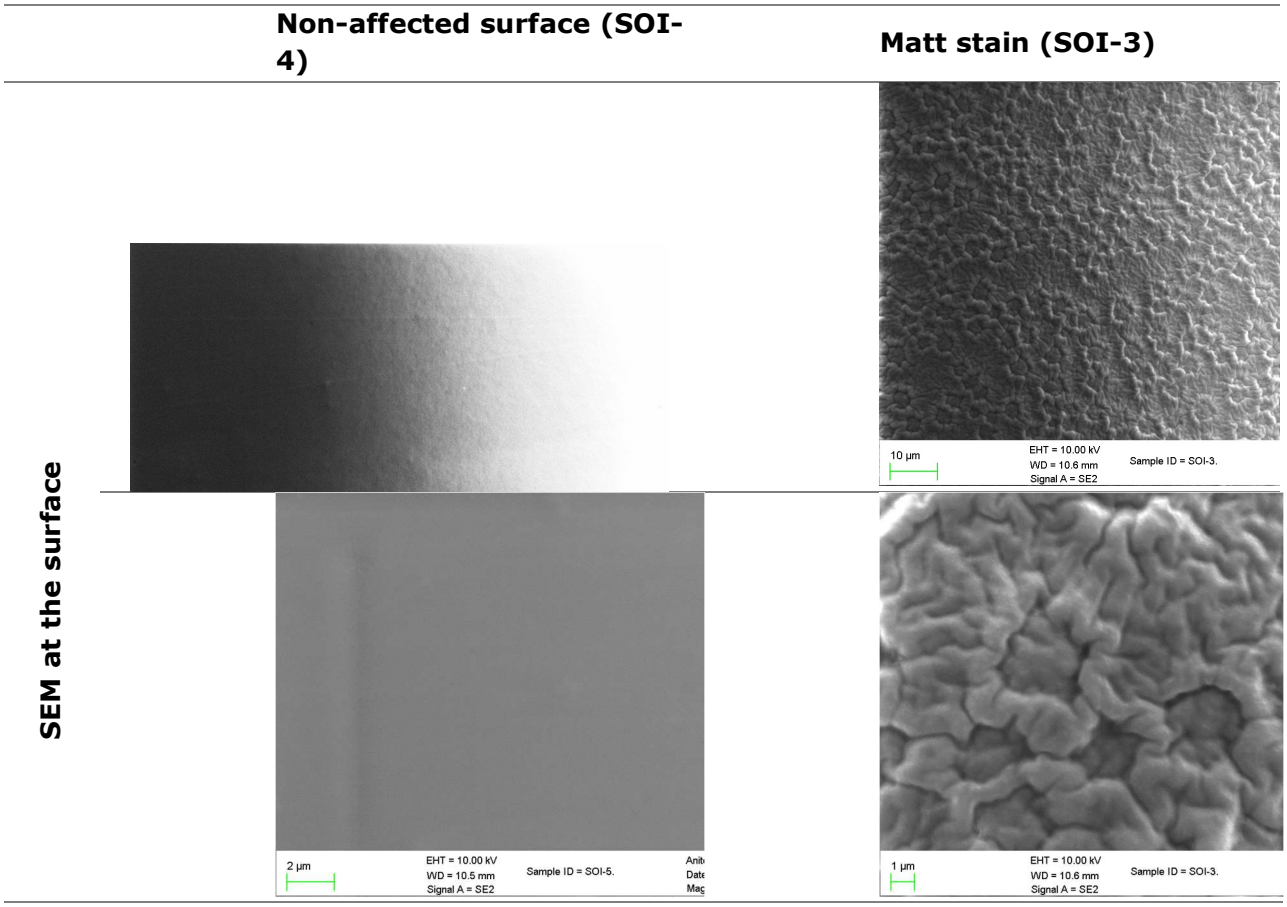


Figure 6 – SEM SE2 images representative of the surface aspect at the non-affected area and matt stain area and FIB cross sectional view and location of the EDS analyses (close to the surface and at two microns depth approximately)

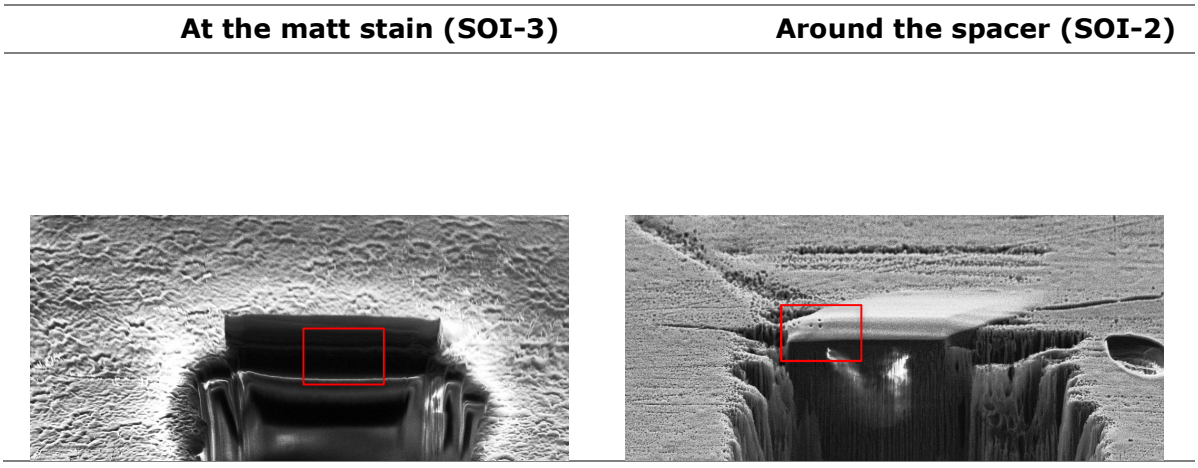


Figure 7 – FIB milling cross section overview in SOI-2 and SOI-3 and location of the images displayed in Figure 8 and Figure 9. The charging effect during the milling caused beam distorsion and alignment issues as it can be observed in the irregular shape of the holes

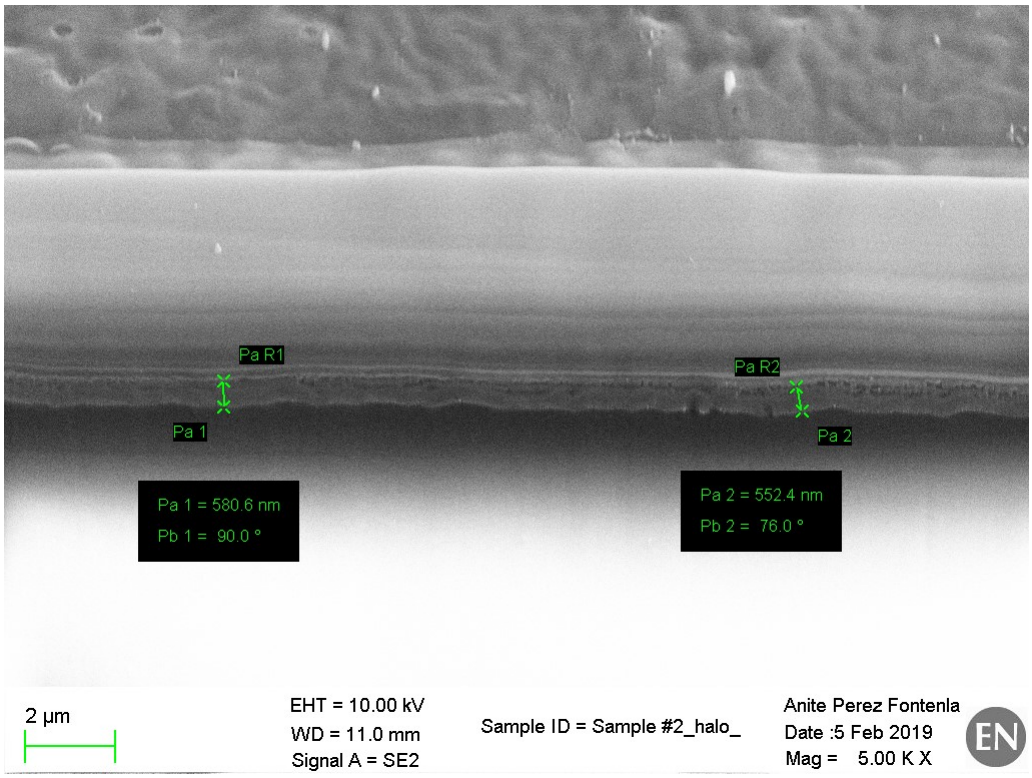


Figure 8 – SEM image detail of the FIB cross section at the surface in SOI-3 (matt stain). A thin layer of approximately 500 nm is noticeable

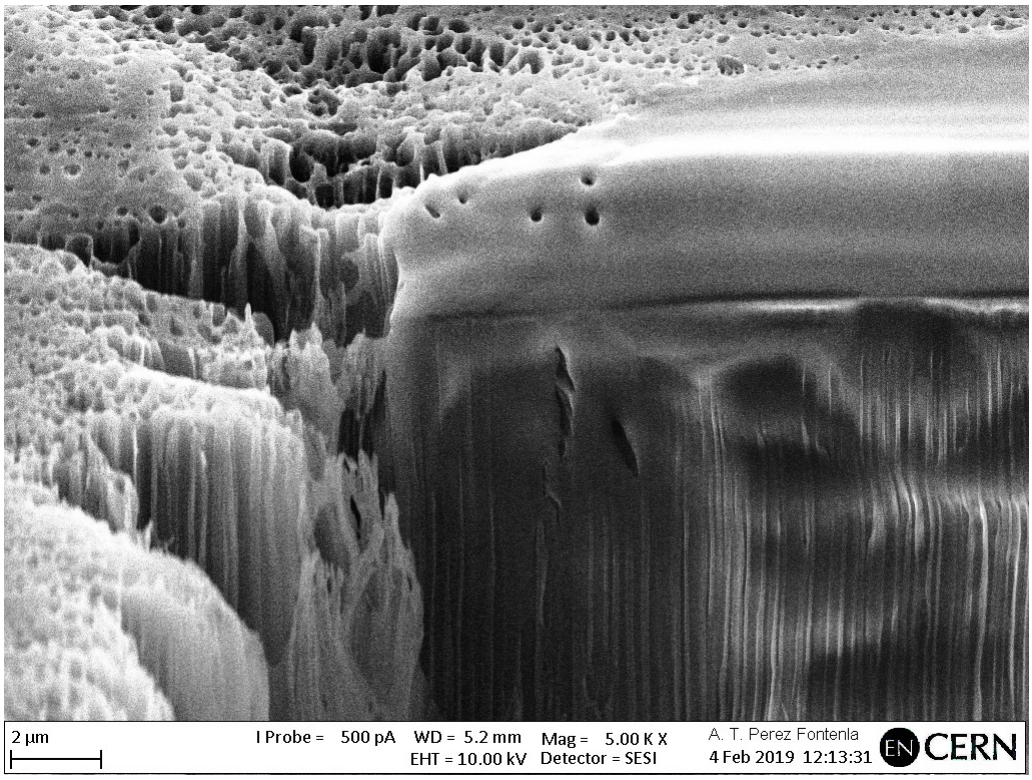


Figure 9 – SEM image detail of the FIB cross section at the surface in SOI-2 (area around the spacer)

3.2 EDS analysis on Sample #4 to Sample #9

Semi-quantitative EDS analysis at 20 keV was performed at various SOI per sample (using the naming described at 2.1) for samples #4 to #9. Summary of the obtained results is presented in Table 2.

Table 2 – Semi-quantitative EDS analysis at 20 keV performed at the different SOI in Sample #4 to Sample #9

Sample ID	SOI	Element wt. % (normalized)	C	N	O	F
Sample #4	4	Bakelite	42.61	34.76	21.95	0
Sample #5	2	Area around spacer	45.39	35.17	17.68	1.6
	3	Halo/Matt stain	69.84	0.76	26.99	2.25
Sample #6	2	Area around spacer	48.71	30.64	14.87	5.63
	3	Halo/Matt stain	70.01	0.91	22.67	6.18
Sample #7	2	Area around spacer	49.77	26.08	18.43	5.55
	3	Halo/Matt stain	68.04	0.85	28.15	2.72
Sample #8	2	Area around spacer	48.4	25.11	26.35	0.1
	3	Halo/Matt stain	67.6	3.24	29.11	0
Sample #9	2	Area around spacer	61.12	9.3	28.84	0
	3	Halo/Matt stain	69.32	2.08	28.14	0

4. Summary of observations

Samples from different gaps were prepared and analysed by means of microscopy techniques (imaging by SE and chemical composition analysis by EDS) at various SOI at the surface and at the cross sections prepared by FIB milling.

- F was detected only on irradiated samples;
- The EDS analysis in Sample #4 (used as reference surface free of linseed oil) showed that the composition is rich in C, N and O. The presence of C and N is well aligned with the composition melamine and C and O are also present on the phenolic resin.
- The EDS analysis in Sample #1 (used as reference surface with linseed oil) showed that the composition at the halo is similar to the composition of the Bakelite without oil (Sample #4).
- Moreover, if we compare the surface aspect by laser interferometry of the samples irradiated in the past that performed well (i. e. Sample #7) with the Sample #2 tested in 2017, thickness inhomogeneity's are visible (study performed by Didier Glaude - EN-MME/MM – EDMS: 2065851).

  Hypothesis: The oil layer is thinner in the halo region leading, after irradiation, to a heavily affected surface.

Sample #7 (irradiated 2010)	Sample #2 (irradiated 2017)

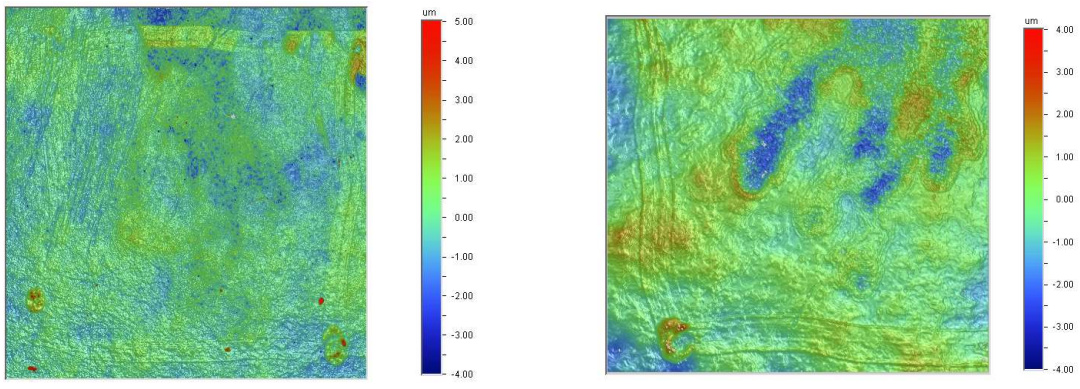


Figure 10 – Laser interferometry results from EDMS 2065851 in Sample #2

- In addition, the microscopy inspection pointed out that matt stains present a certain roughness in opposition to non-affected areas and non-irradiated samples that present a relatively smooth surface;

5. Annex

Sample ID	SOI	Element wt. % (normalized)	C	N	O	F
Sample #1	1	Spacer glue area	75.21	4.37	19.02	0
	2	Area around spacer	42.84	45.86	10.53	0
	3	Halo/matt stain	54.4	24.82	20.09	0
	4	Non-affected area	42.84	48.7	7.99	0
Sample #2	1	Spacer glue area	77.3	0.0	20.6	1.5
	2	Area around spacer	44.0	41.6	13.0	1.4
	3	Halo/matt stain	70.4	0.0	27.3	2.3
	4	Non-affected area	50.1	28.4	18.9	2.7
Sample #3	1	Edge glue area	74.6	3.6	19.6	1.7
	2	Halo/matt stain	42.1	47.9	9.7	0.2
Sample #4	4	Bakelite	42.61	34.76	21.95	0
Sample #5	1	Spacer glue area	42.24	43.29	14.21	0.13
	2	Area around spacer	45.39	35.17	17.68	1.6
	3	Halo/matt stain	69.84	0.76	26.99	2.25
Sample #6	2	Area around spacer	48.71	30.64	14.87	5.63
	3	Halo/matt stain	70.01	0.91	22.67	6.18
Sample #7	2	Area around spacer	49.77	26.08	18.43	5.55
	3	Halo/matt stain	68.04	0.85	28.15	2.72
Sample #8	2	Area around spacer	48.4	25.11	26.35	0.1
	3	Halo/matt stain	67.6	3.24	29.11	0
Sample #9	2	Area around spacer	61.12	9.3	28.84	0
	3	Halo/matt stain	69.32	2.08	28.14	0