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Microscopic examination of triple-GEM detector after installation and operation on LHCb with CF_4-based gas mixture

Summary:

Previous studies in 2004 showed that after operation with CF_4-based gas mixture, the copper surface of a triple-GEM detector was etched due to bad gas flow rate conditions [1]. The aim of the current study is to inspect by microscopic means different layers of a triple-GEM detector after its operation in LHCb also with CF_4-based gas mixture.

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1. Introduction

Previous studies in 2004 showed that after operation with CF_4-based gas mixture, the copper surface of the triple-GEM detector was etched due to bad gas flow rate conditions [1].

1.1 Aim of the study

The aim of the current study is to inspect by microscopic means the different layers of a triple-GEM detector after its operation in LHCb also with CF_4-based gas mixture.

1.2 Key words

OM, SEM, FIB-SEM, EDS, CF4, Gas Electron Multiplier, GEM, LHCb

2. Protocol

2.1 Samples

The samples under study are part of a triple-GEM detector after operation in LHCb with CF_4-based gas mixture (40% CF_4, 15% CO_2, 45% Ar). The detector is composed by three rectangular GEM layers consisting on a thin, copper-cladded Kapton foil, chemically pierced by holes with a nominal diameter of 70 microns. The layers are connected to a glass-fibre frame and the entire component is closed/sealed by glue. In the present report, the results on the following layers are presented:

- GEM#1: Bottom layer on the detector;
- GEM#3: First accessible layer of the detector as received. During visual inspection some colouring was noticed especially evident on the gas outlet region.

Optical microscopy was performed on both layers while mounted on the frame. To access the bottom layer (GEM#1) the edges of the upper ones were cut with a metallic tool. For SEM observation, smaller specimens were cut from each layer using scissors.

2.2 Equipment

- Digital microscope KEYENCE VHX 6000
- Field Emission Gun Scanning Electron Microscopes (FEG-SEM) Sigma and Sigma 500 (from ZEISS) with InLens Secondary Electron (SE), Everhart-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 for imaging.
- 50 mm^2 X-Max Energy Dispersive X-Ray Spectroscopy (EDS) detector and AzTEC software (from Oxford Instruments) for chemical analysis;
- EDS X-Max Extreme Windowless 100 mm^2 detector (from Oxford Instruments) for high-sensitivity chemical analysis at low voltage;

- EDS detection: Makes impossible to detect presence of elements below around 0.2 wt. % (the value dependents on the relative weight of the different elements), or light elements (impossible below Z=2 for Extreme detector).

3. Results and discussion

3.1 GEM#1

The general aspect of the GEM#1 is homogenous. Nevertheless, when observed on the microscope some features and colour contrast on the copper (Cu) surface were visible. Three zones were identified as shown in Figure 1 and imaged by optical and electron microscopy. The red squares correspond to areas inspected at higher magnification (zones A.1, A.2, B and C).

It was noticed that the Cu surface surrounding numerous holes present an oxidized aspect. The SEM observation showed that they correspond to holes where the Cu edges were damaged during operation (molten aspect) as shown in Figure 3.

Also remarkable all over the Cu surface on GEM#1 is the presence of micrometric marks. When observed by SEM they correspond to sites where the Cu was attacked or removed up to a depth of ~ 1 micron (see Figure 4 and Figure 5). EDS does not show major compositional differences between the marks and non-affected Cu (Figure 6).

Chemical analysis by EDS was also performed on the hole's surrounding area and it was observed that the content of sulphur (S), nitrogen (N) and oxygen (O) slightly increase when approaching the hole edge. Also the carbon (C) increases due most probably to the Kapton presence and fluorine (F) remain constant and in all cases below 0.1 wt. % as presented in Figure 8.

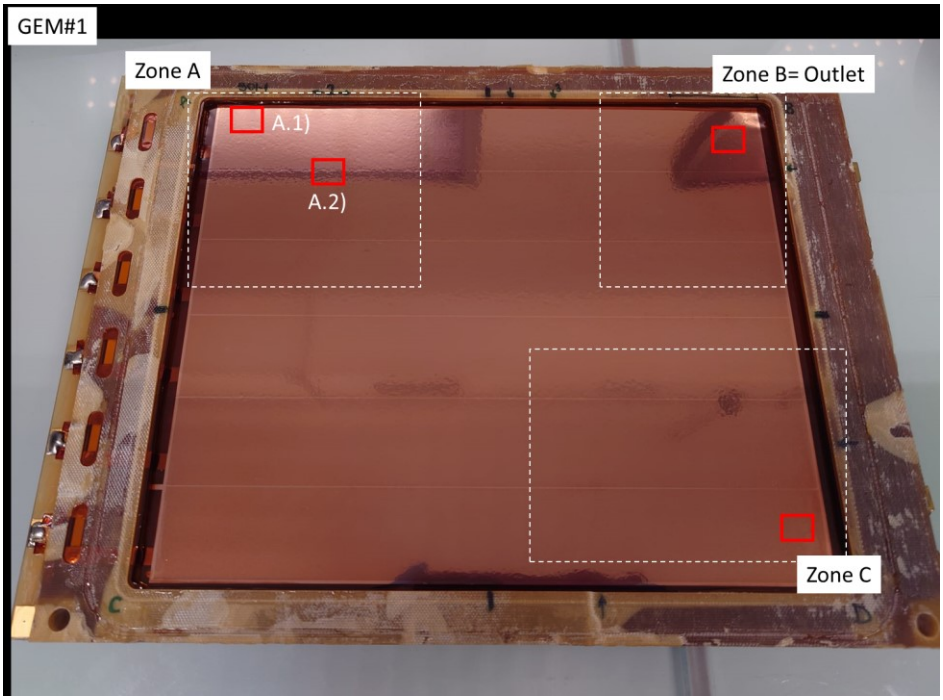

Figure 1 - General aspect and areas under study on GEM#1

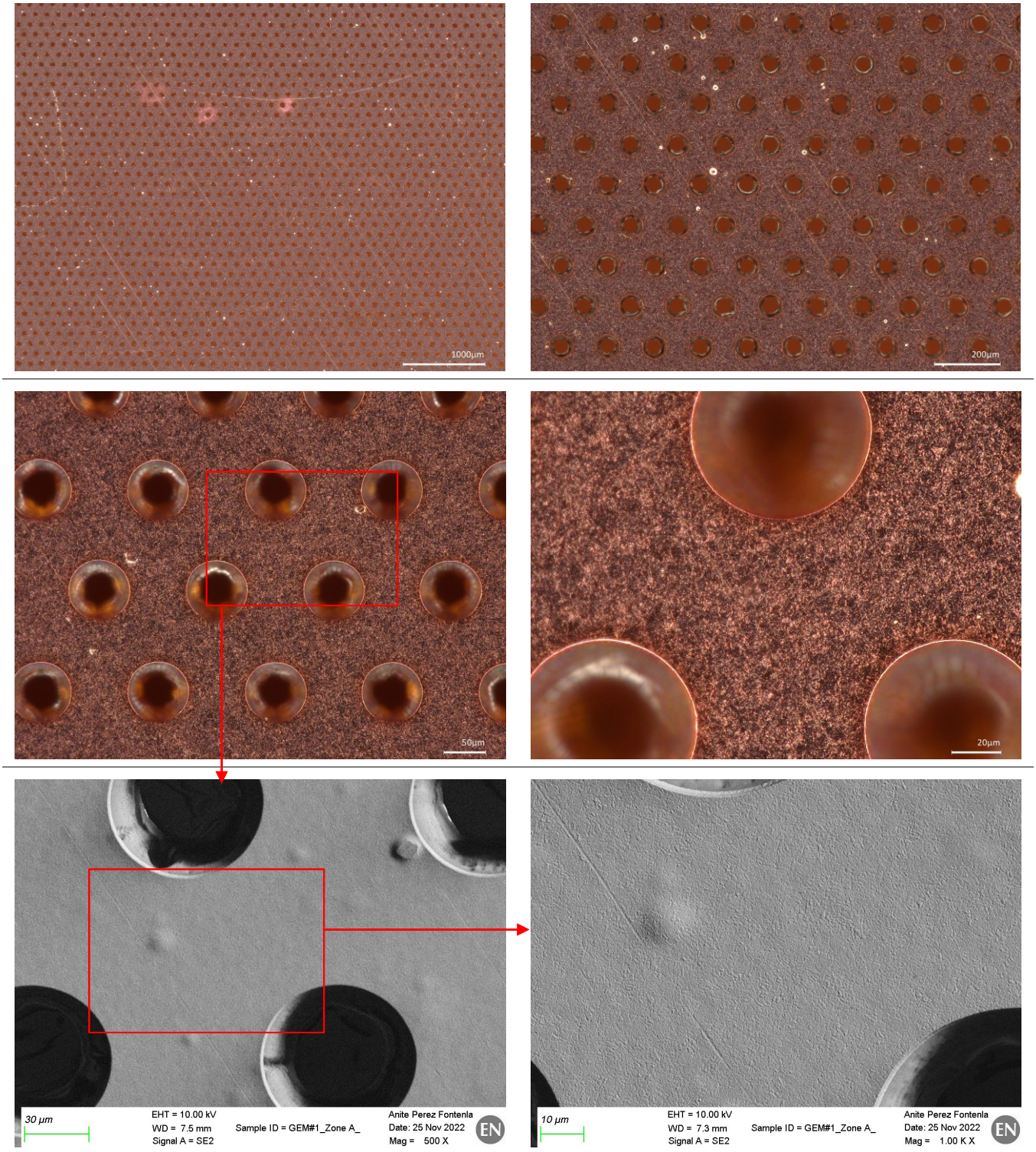

Figure 2 - OM and SEM images of the GEM#1 aspect on zone A.1

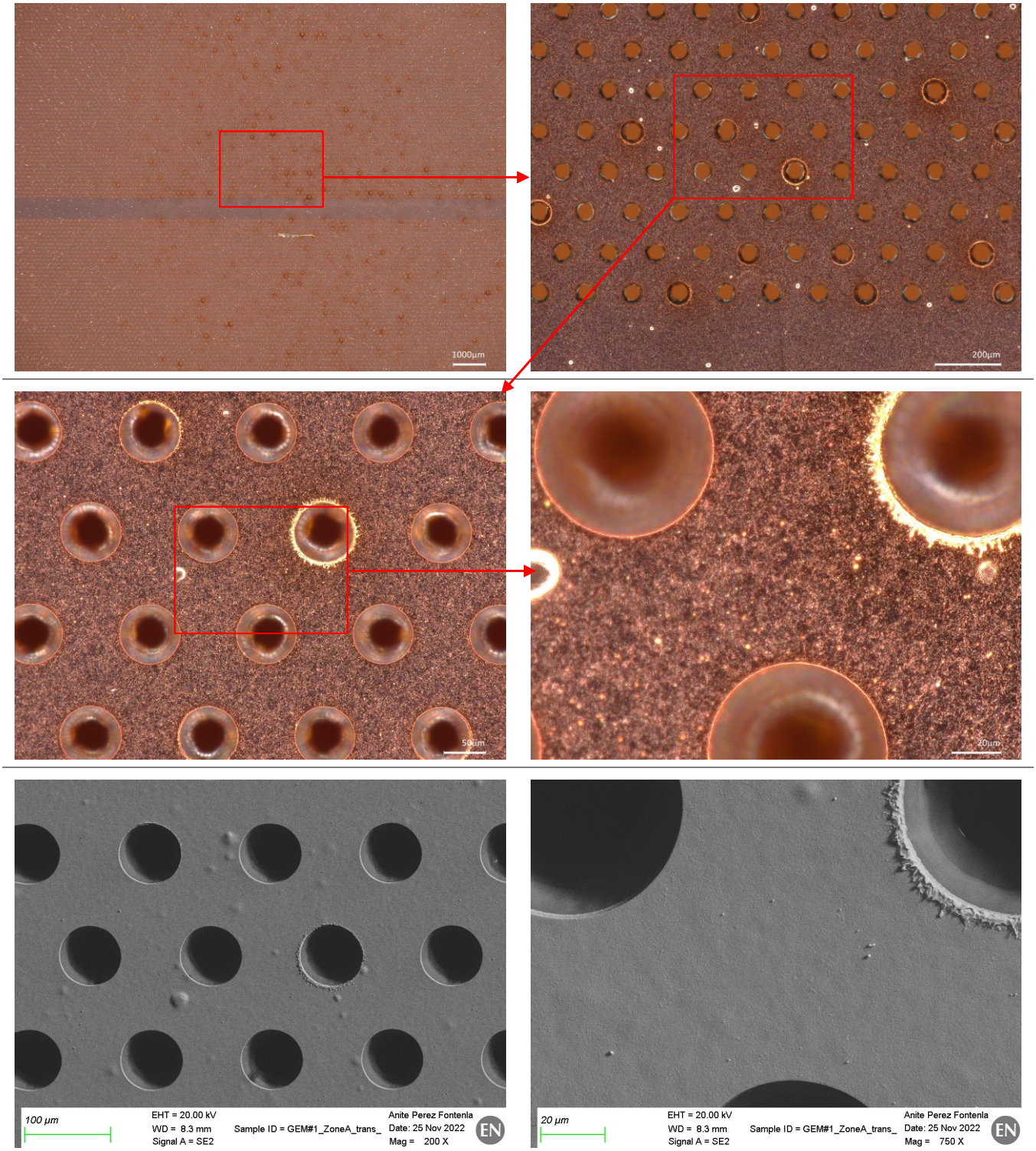

Figure 3 - OM and SEM images of the GEM#1 aspect on zone A.2

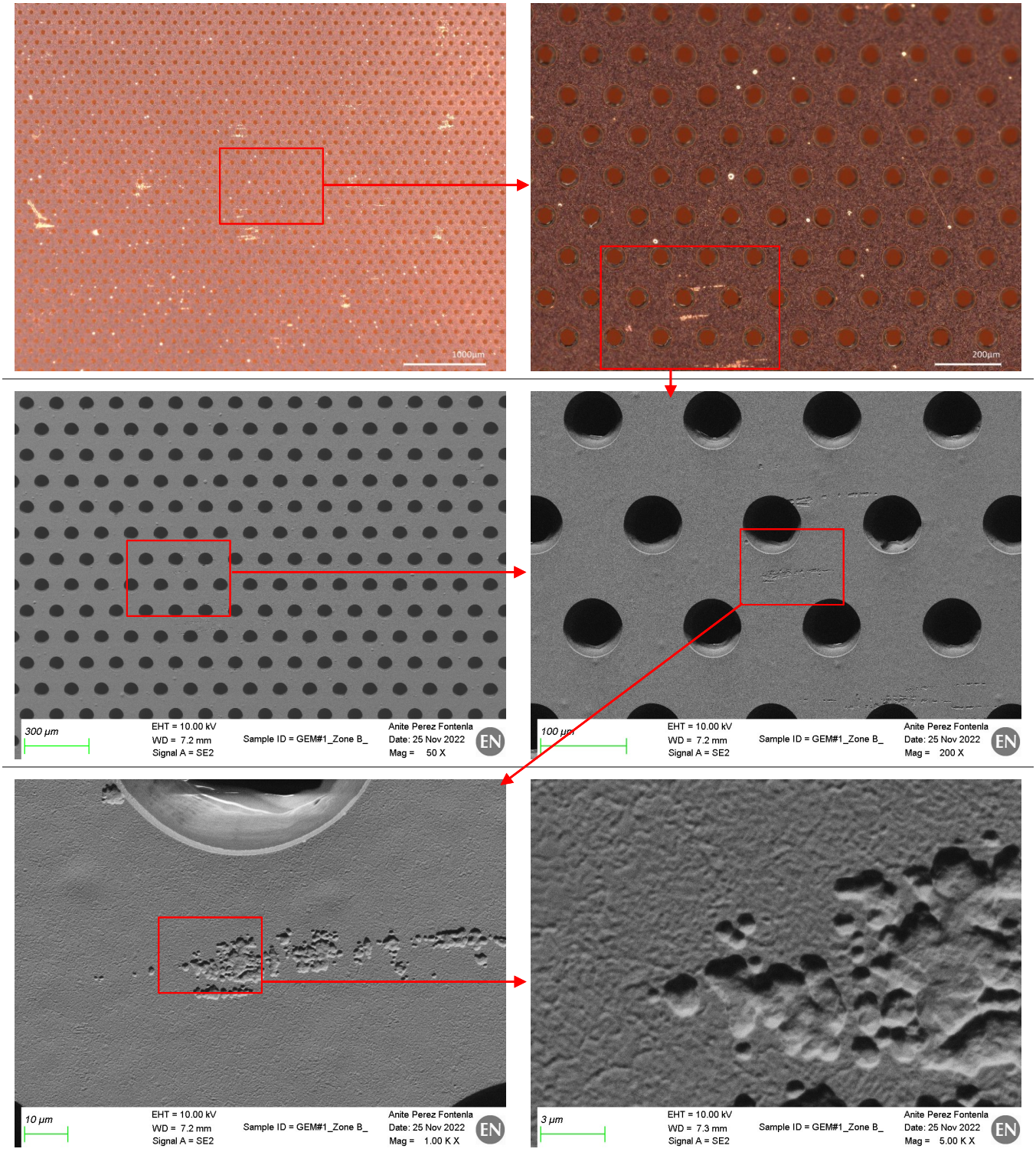

Figure 4 - OM and SEM images of the GEM#1 aspect on zone B (top view)

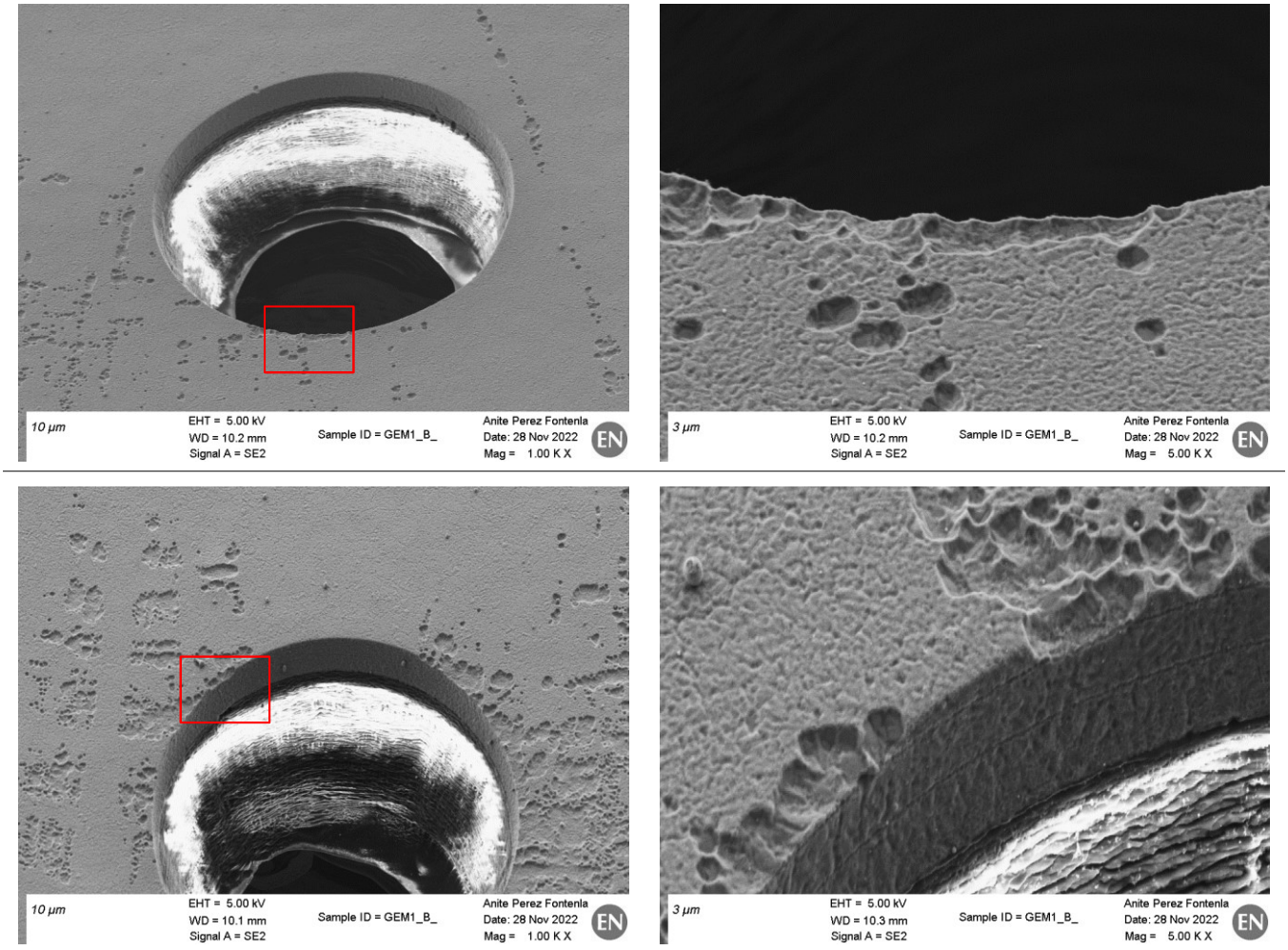

Figure 5 - SEM images of the GEM#1. Cu features detail around holes on zone C (sample tilted 45 degrees)

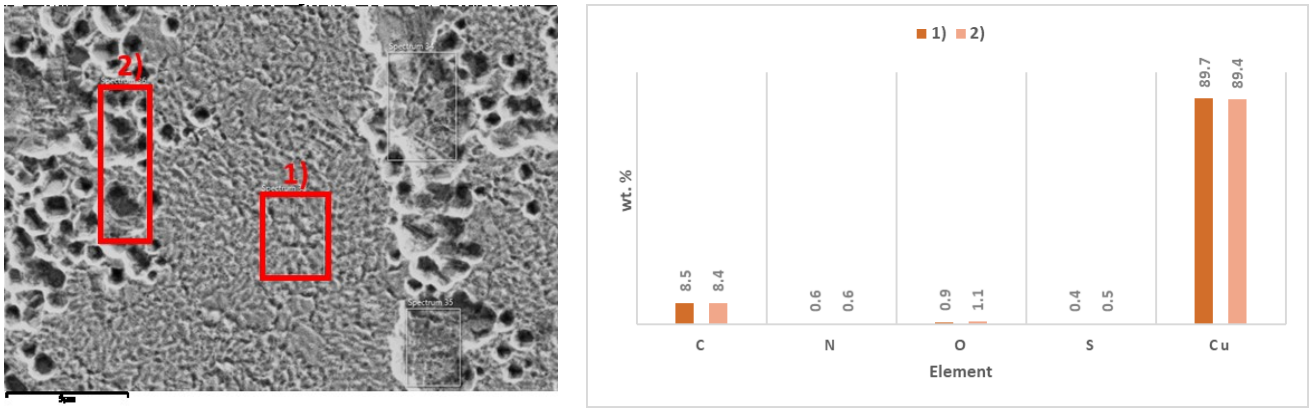

Figure 6 – Elemental composition analysis by EDS on attacked site (GEM#1). The results are presented in wt. % and normalized

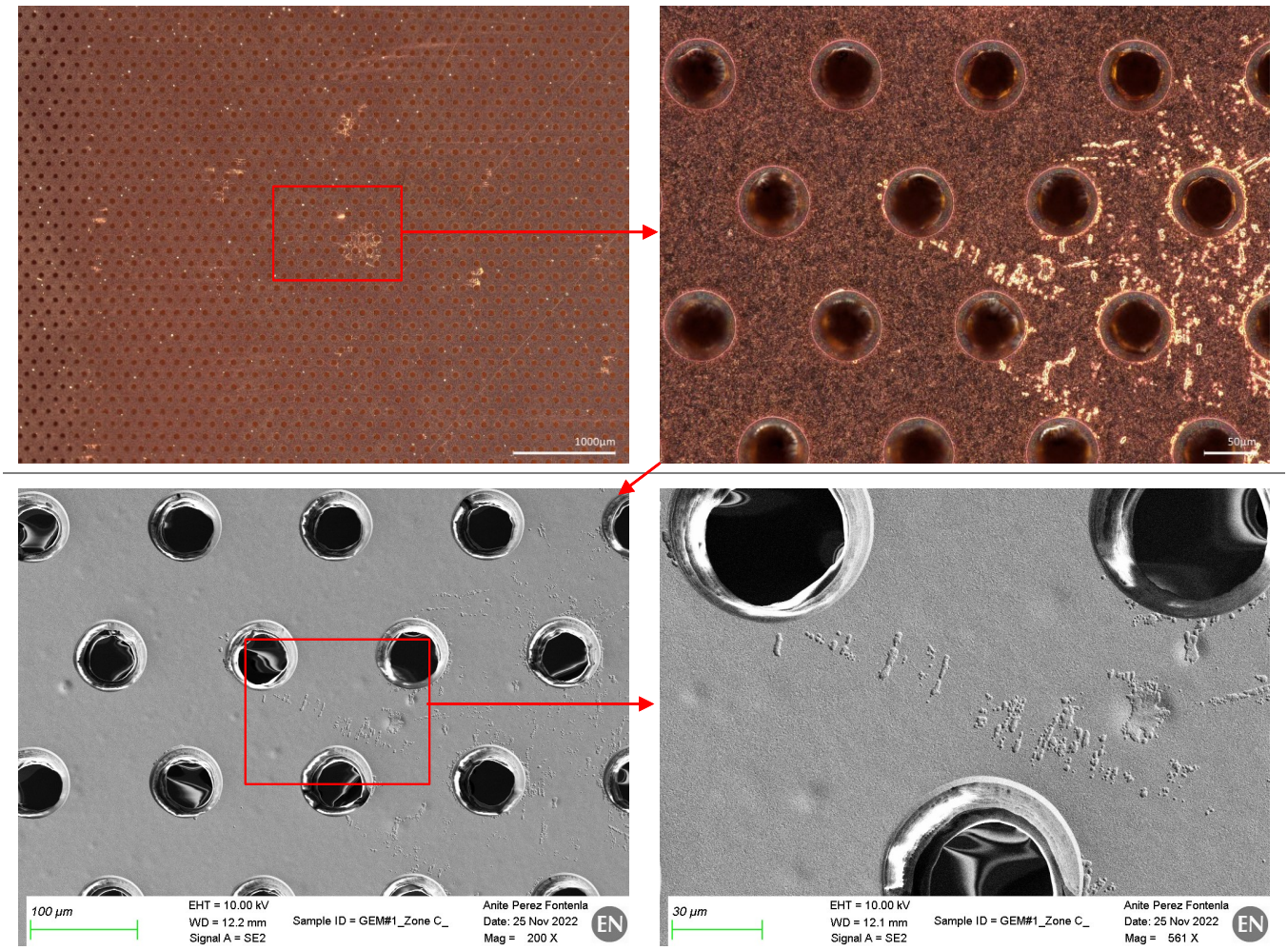

Figure 7 - OM and SEM images of the GEM#1 aspect on zone C

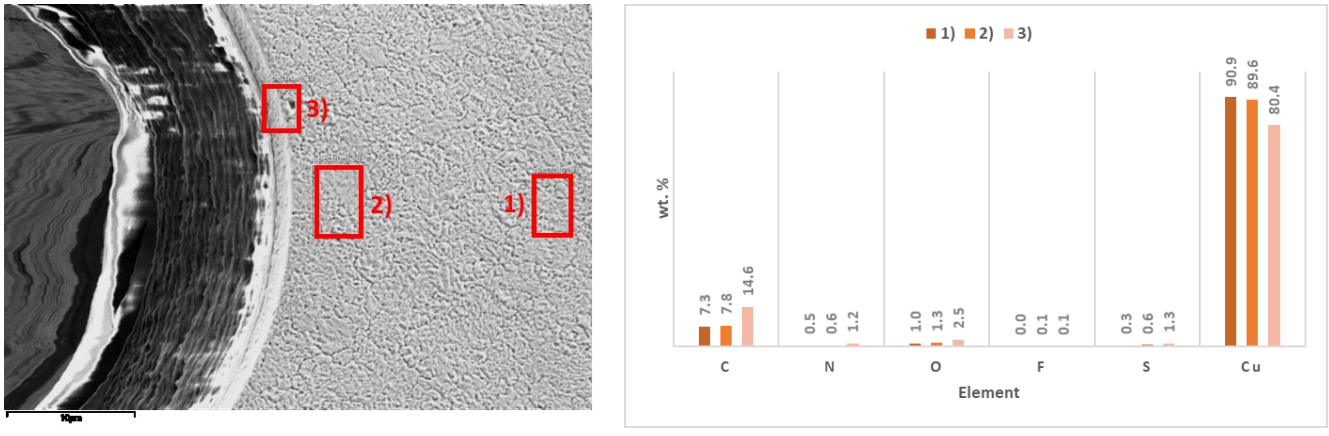

Figure 8 – Elemental composition analysis by EDS on the hole surrounding area on zone B (GEM#1). The results are presented in wt. % and normalized

3.2 GEM#3

Digital microscope inspection was performed on the different areas that presented colour contrast. The location of the studied areas is shown in Figure 9 (zones A.1, A.2, A.3, B and C).

In location A.1, the Cu surface colour around the holes changes concentrically. In addition, micrometric particles are visible surrounding the holes (see Figure 10). SEM inspection performed in location A.1 and A.2 confirmed the presence of the micrometric particles. The hole's edges appear rougher as well as the Kapton into the holes (see Figure 11).

Chemical analysis by EDS on the hole's surrounding areas pointed out an increasing content of C, S, N and F when approaching the hole's edge as shown in Figure 12.

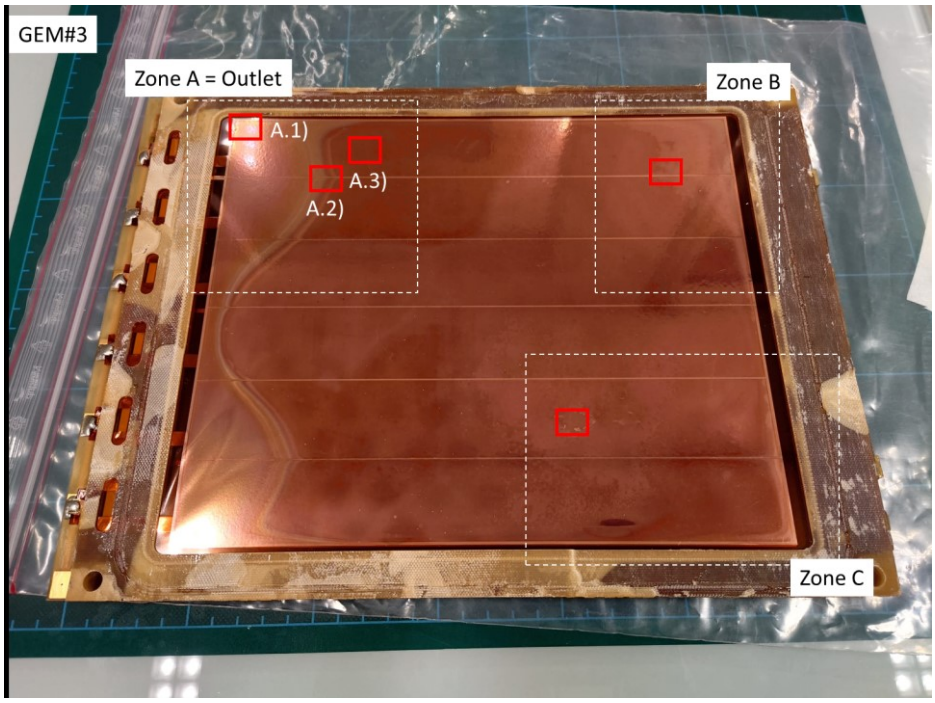

Figure 9 – General aspect and areas under study on GEM#3. The red squares correspond to areas inspected at higher magnification

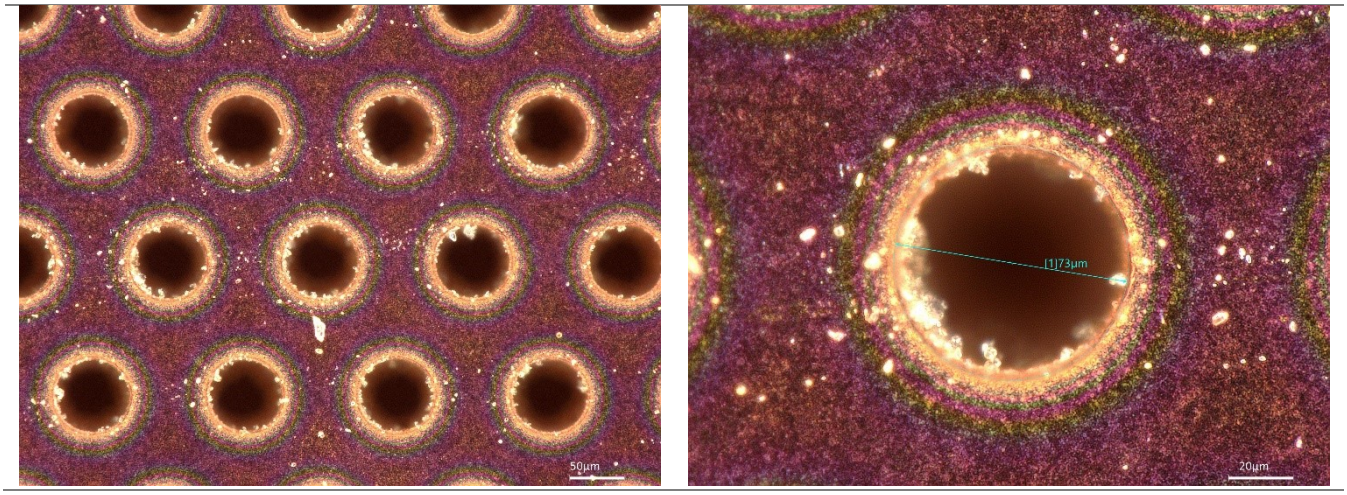

Figure 10 – OM images of the GEM#3 aspect on location A.1

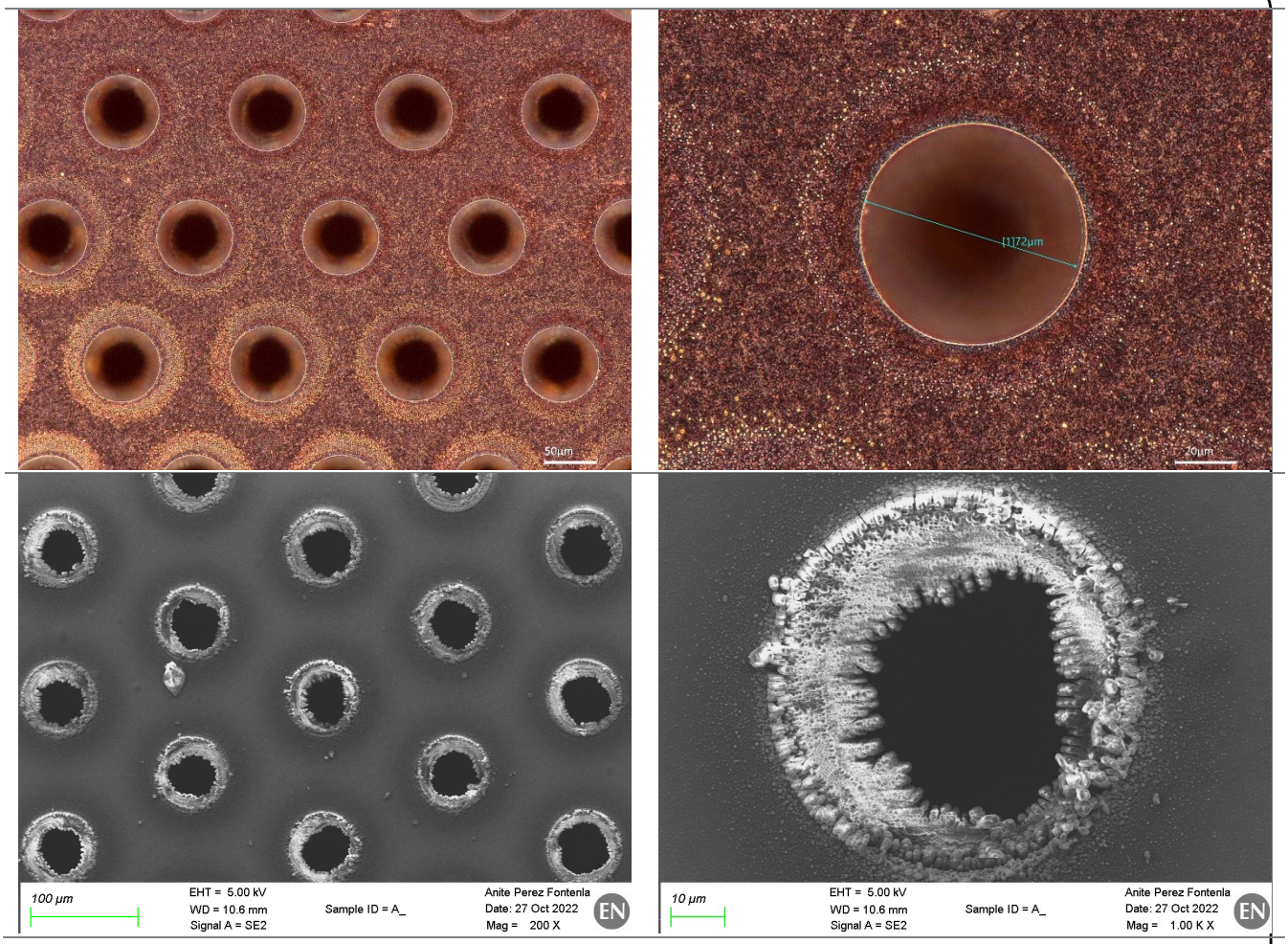

Figure 11 – OM and SEM images of the GEM#3 aspect on zone A.2

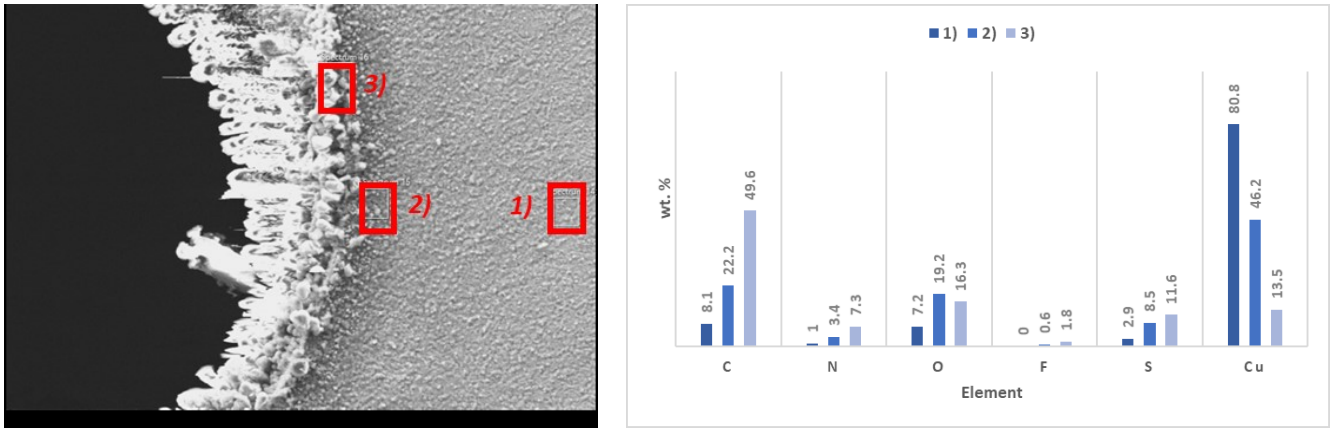

Figure 12 – Elemental composition analysis by EDS on the hole surrounding area on zone A (GEM#3). The results are presented in wt. % and normalized

FIB cross section (of the full layer thickness) was prepared with a current of 100 nA and accelerating voltage of 30 keV. Subsequent milling steps at 60 nA, and 15 nA (with accelerating voltage of 30 keV) were then performed to leave an enough smooth surface that could be effectively imaged and analysed. Comparison of the three sites is shown in Figure 13.

FIB-SEM inspection confirmed that the residue observed on the hole's edges is also deposited into the hole internal surface covering the Kapton and modifying the hole's geometry. The layer is thicker on location A.1 (~ 2 µm), is also present but thinner in location A. 2 (~ 1 µm) and is just noticeable in location A.3 (nanometric).

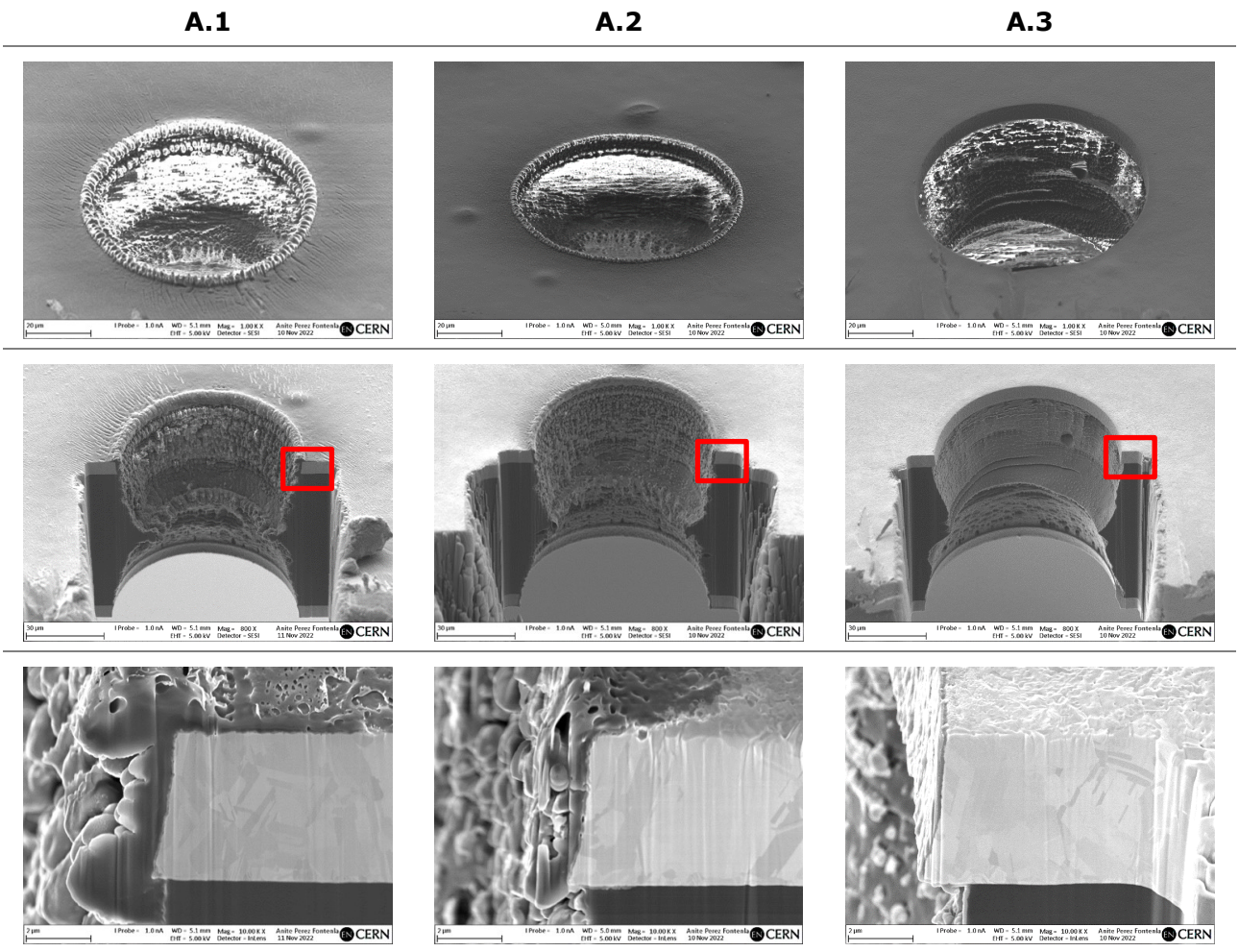

Figure 13 – Representative SEM images of holes in locations A.1), A.2) and A.3) before and after FIB cross sectioning and detail of the deposit on the copper edge

A more detailed observation on the hole cross section on site A.1 and a comparison of the EDS spectrum on the deposit and the Kapton are included in Figure 14. The deposit presents a significant amount of S and F when compared with the Kapton.

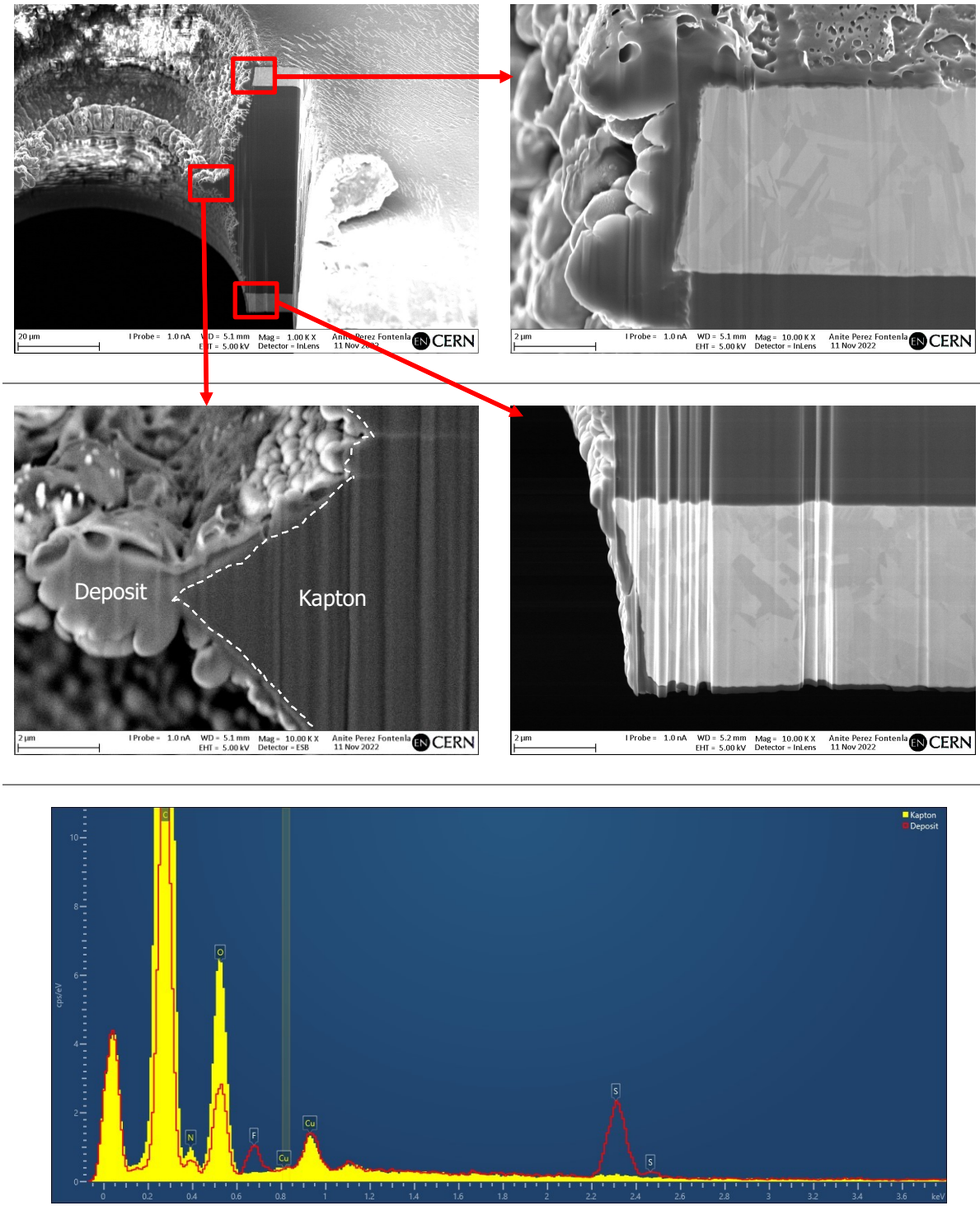

Figure 14 – SEM images of the hole cross section on GEM#3 location A.1 and comparison of the EDS spectra on the deposit and the Kapton

Microscopic observation on zone B and C confirmed the presence of a residue visible on the Cu surface. Representative images are included in Figure 15 and Figure 16 respectively. The chemical analysis of the deposit confirmed the same composition as observed in zone A with

presence of C, O and S. In some cases, traces of F were also detected. Comparison of EDS results on the residue and cleaner sites is included in Figure 17.

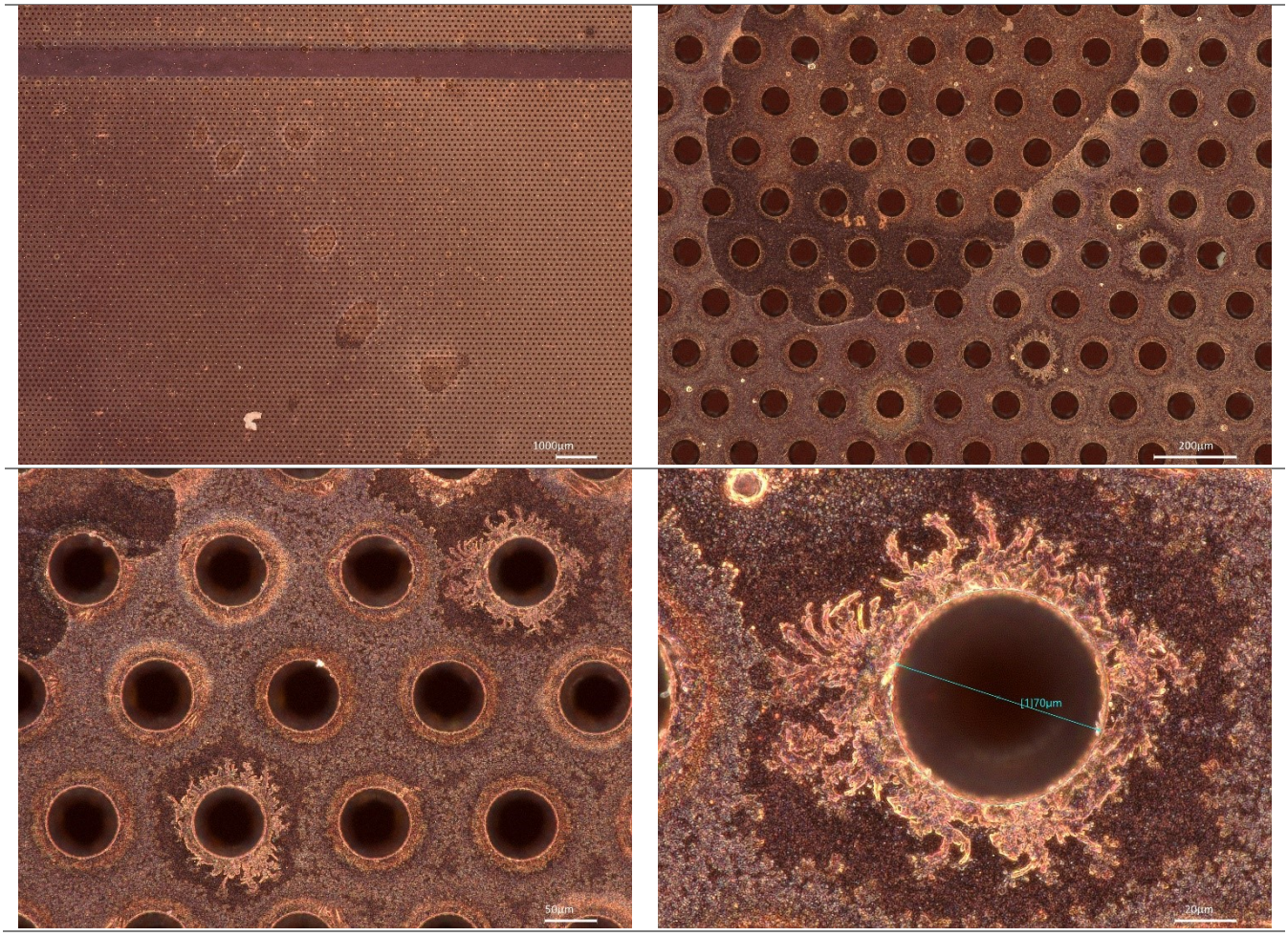

Figure 15 – OM images of the GEM#3 aspect on zone B

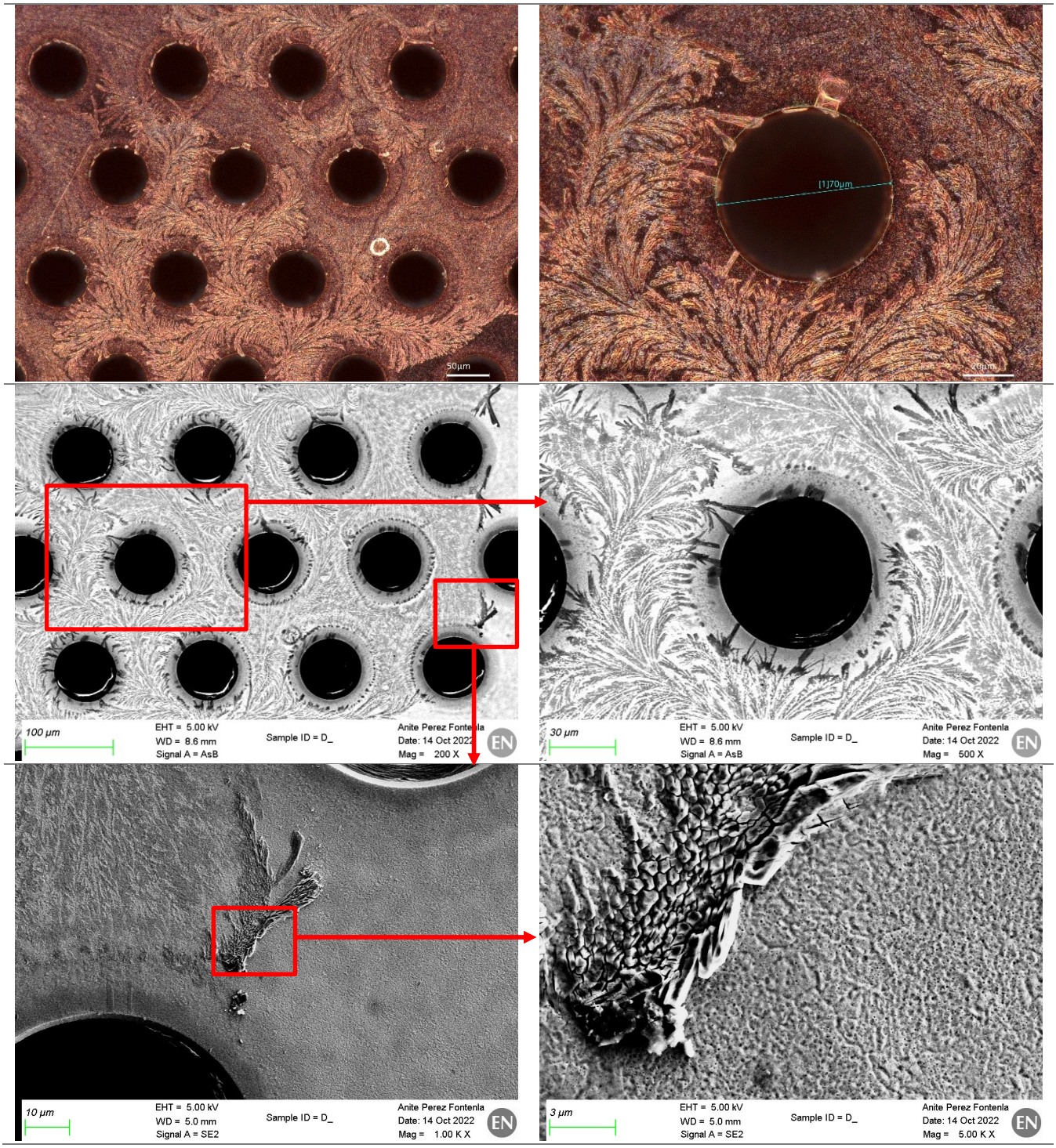

Figure 16 – OM images of the GEM#3 aspect on zone C

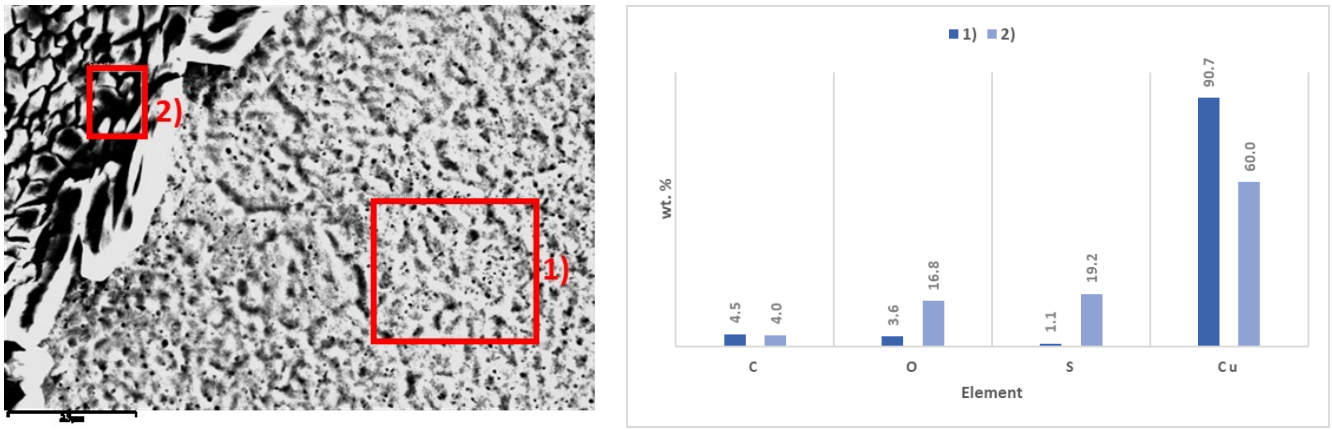

Figure 17 – Elemental composition analysis by EDS on the residue on zone C (GEM#3). The results are presented in wt. % and normalized

4. Summary of observations

Surfaces of GEM#1 and GEM#3 of a triple-GEM detector were inspected by optical and electron microscopy after installation and operation in LHCb.

GEM#1:

- The general aspect of the layer is homogenous;
- Numerous holes randomly located presented an oxidized aspect of the surrounding Cu. They correspond to holes where the Cu edges were damaged during operation (molten aspect);
- Micrometric marks are visible all over the Cu surface. When observed by SEM they correspond to sites where the Cu was attacked or removed up to a depth of ~ 1 micron. They are randomly distributed and present certain orientation. EDS does not show major compositional differences between the marks and non-affected Cu;
- In general, the Cu surface presented contamination of other elements like S, N and in some cases traces of F. The S origin is unknown and it is also present on the glass fibre frame (glued regions).

GEM#3:

- Different areas presented colour contrast during visual inspection;
- The analysis confirmed the presence of a residue all over the GEM surface with significant presence of S (in some areas ~ 20 wt. %);
- In zone A (outlet), the residue was also deposited on the hole's edges and into the hole internal surface covering the Kapton and modifying the hole's geometry. The layer thickness varies from few nanometers to ~ 2 µm.

5. References

[1] M. A. e. al., "Studies of Etching Effects on Triple-GEM Detectors Operated With CF4-Based Gas Mixtures," *IEEE TRANSACTIONS ON NUCLEAR SCIENCE,* vol. 52, pp. 2872-2878, 2005.