Microscopy investigation of the surface behaviour of different materials after H^- irradiation in different conditions

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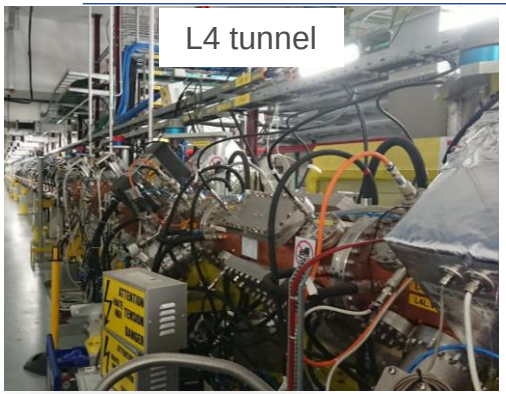
on behalf of the spare RFQ Project team

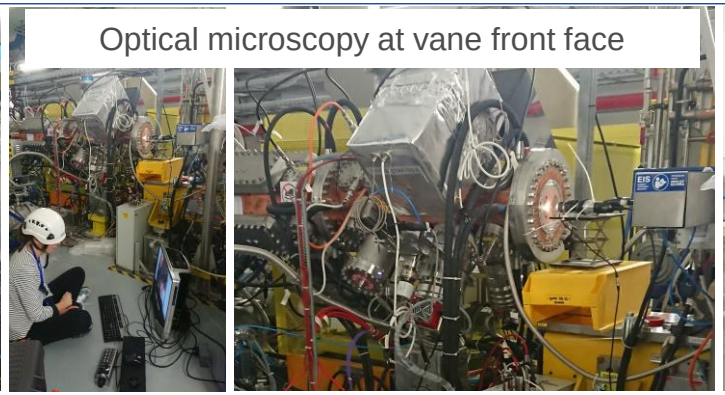
CERN | EN ENGINEERING DEPARTMENT

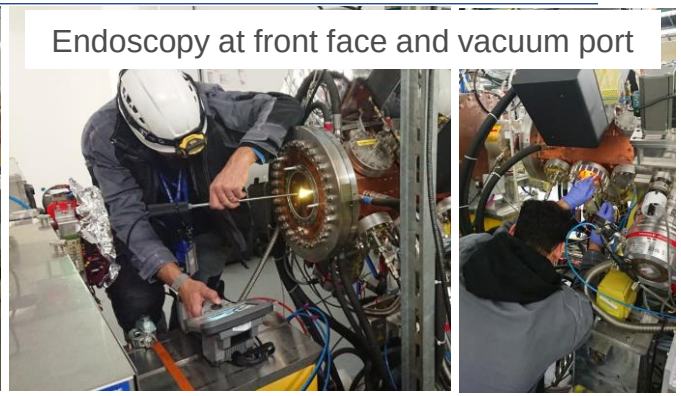
Outline

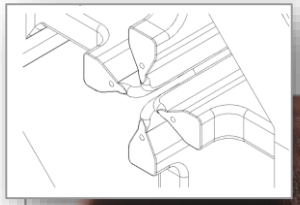
1. Linac4 RFQ in-situ inspection
2. Additional study on collimator
3. Proposed studies
4. Results
5. Summary

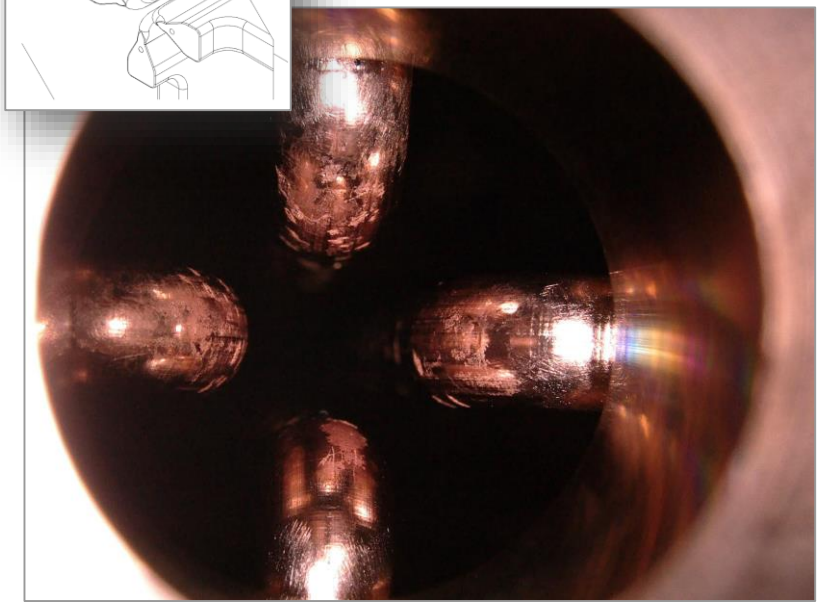
Linac4 RFQ in-situ inspection

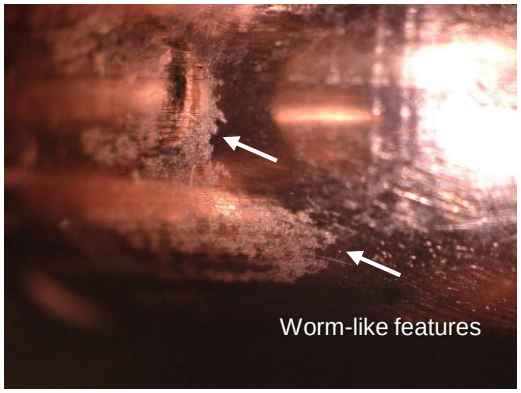


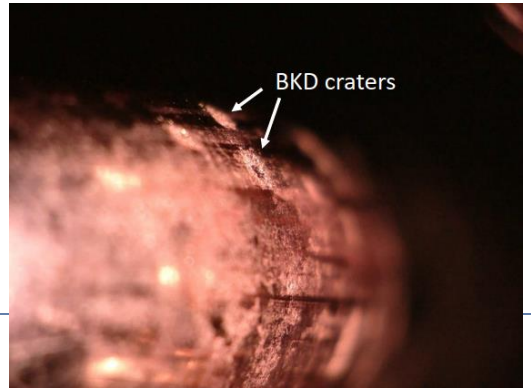




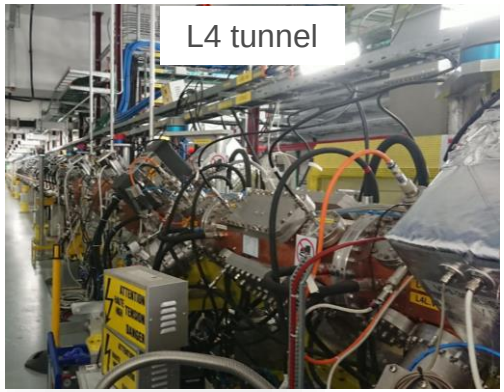


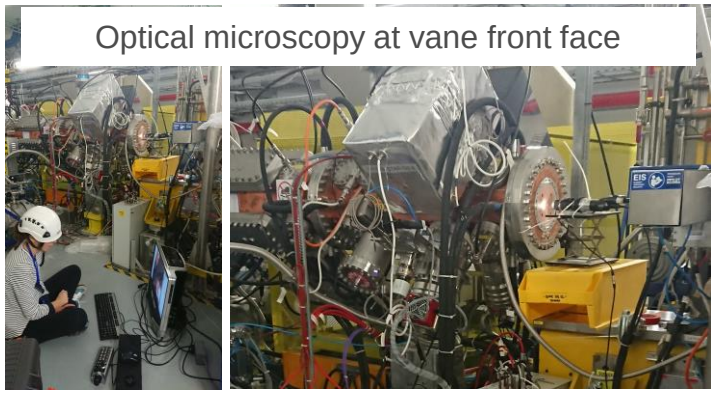


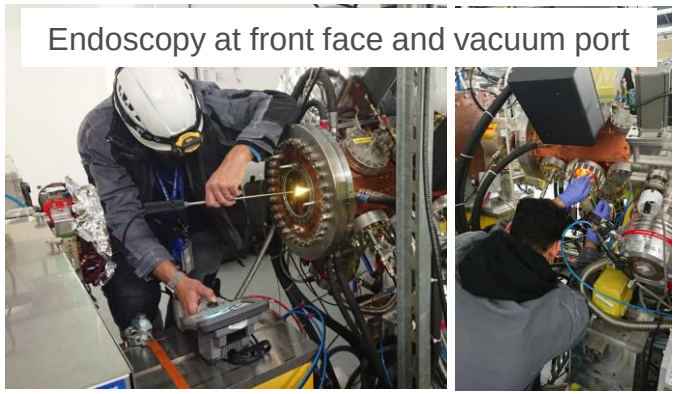


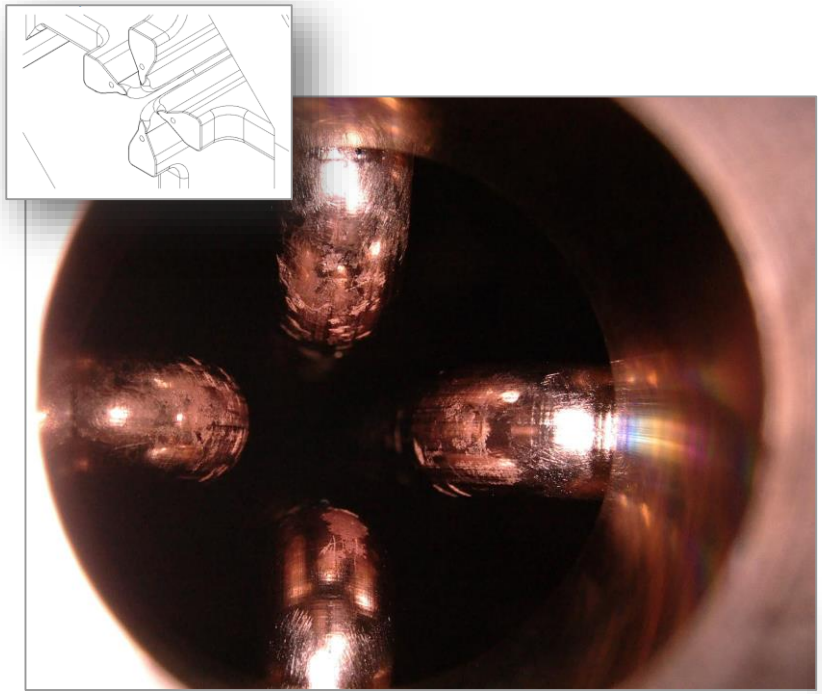


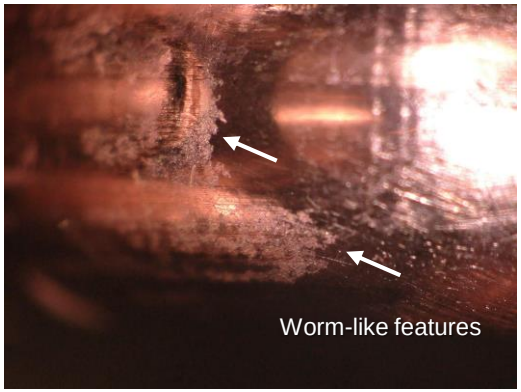
Linac4 RFQ in-situ inspection

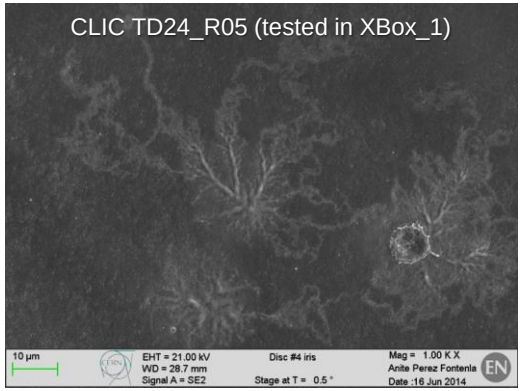


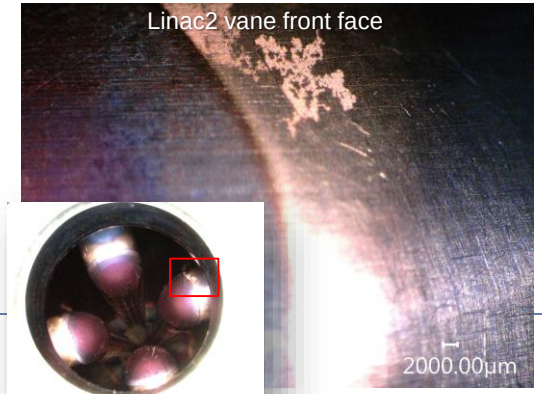


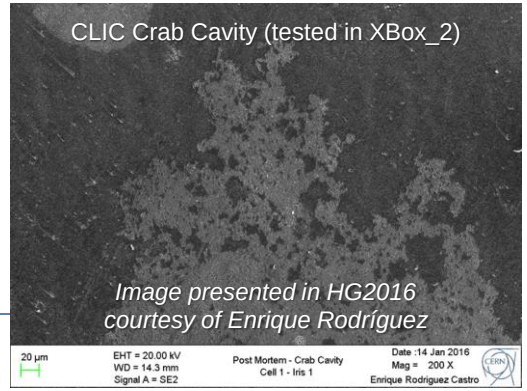




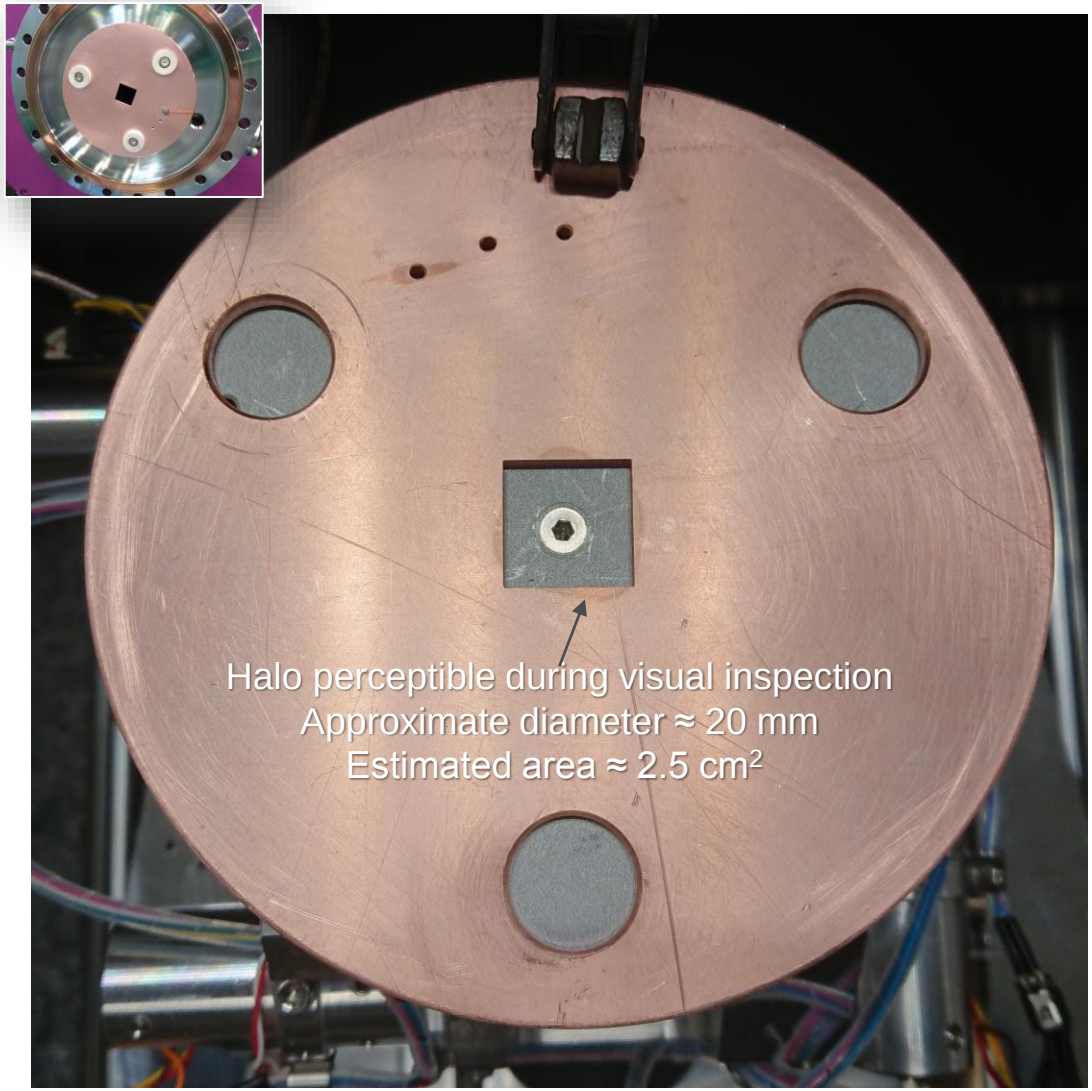






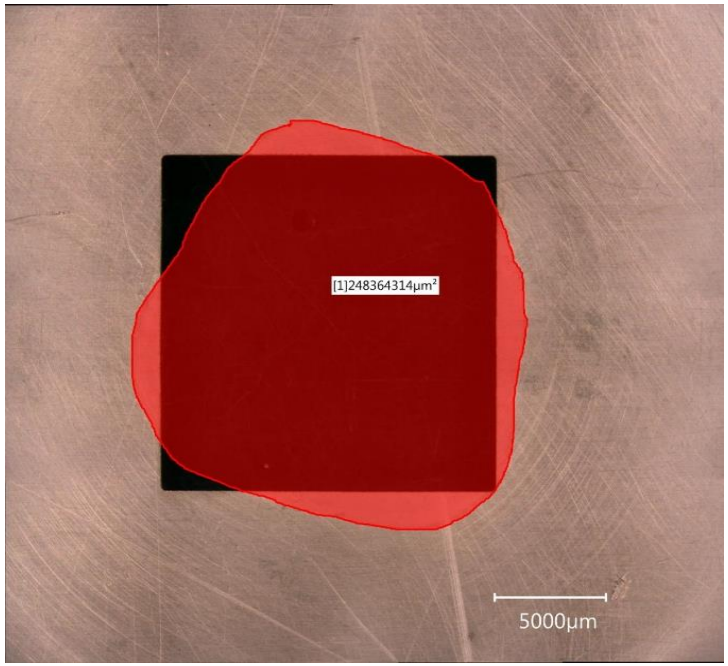


Additional study on Cu-OFE collimator



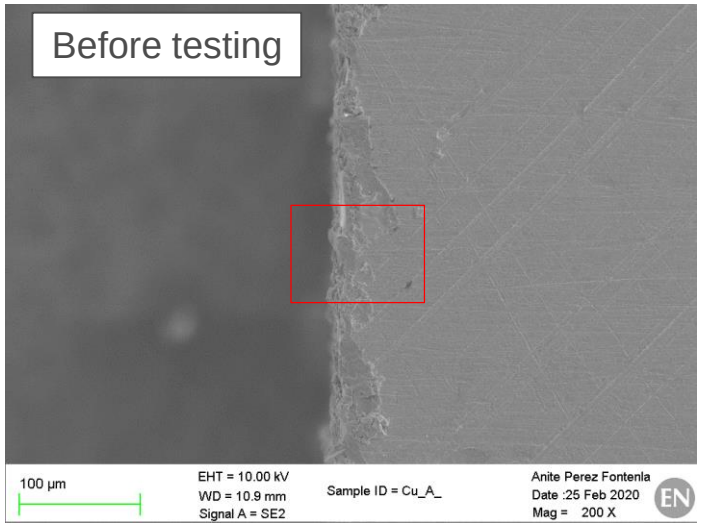
Collimator/mark fabricated from cold rolled Cu-OFE plate (3 mm thickness) was installed and tested at 1.7 x 10^{19} H^- ions at 45 keV (number of total impacting ions calculated from the electrical signal)

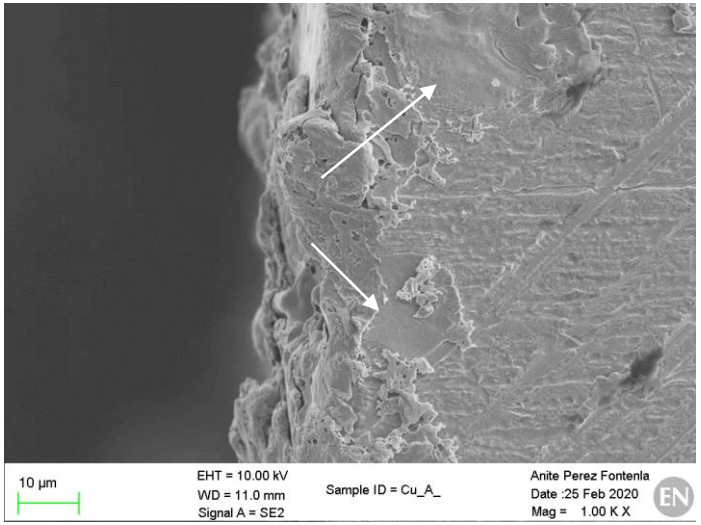
Microscope inspection of the Cu surface around the square was performed before and after testing

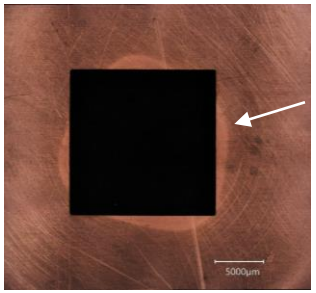


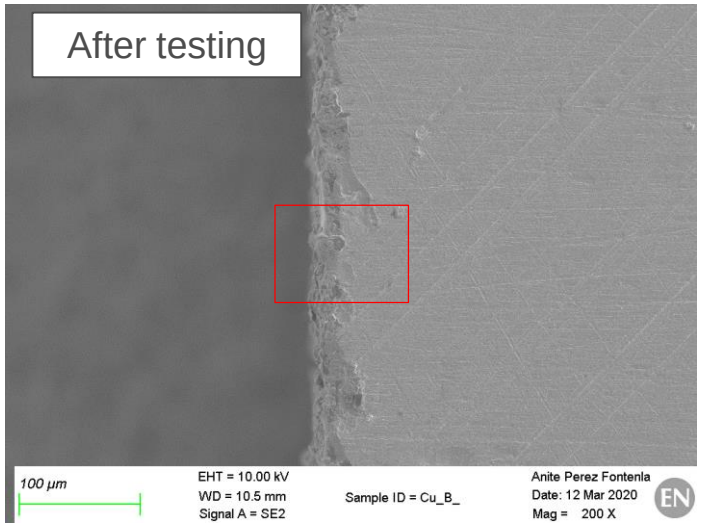
Additional study on Cu-OFE collimator

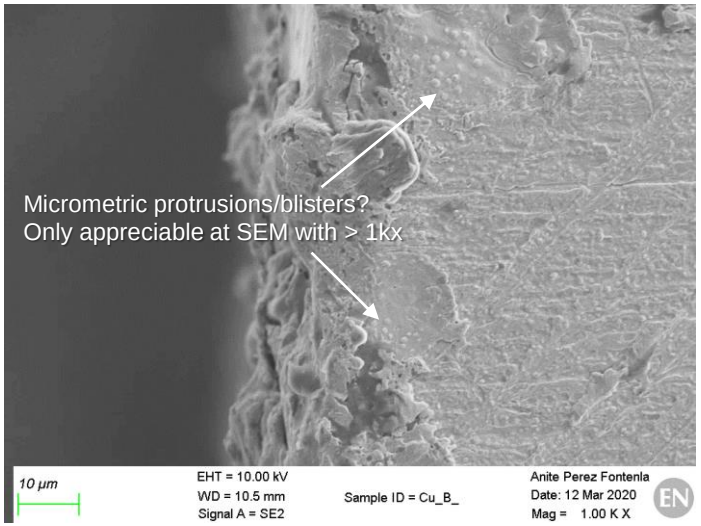
Comparison before and after testing pointed out the presence of blisters



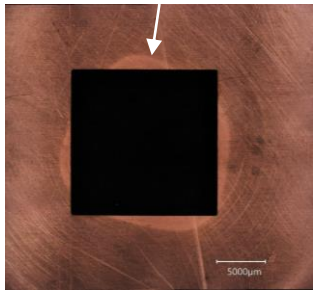








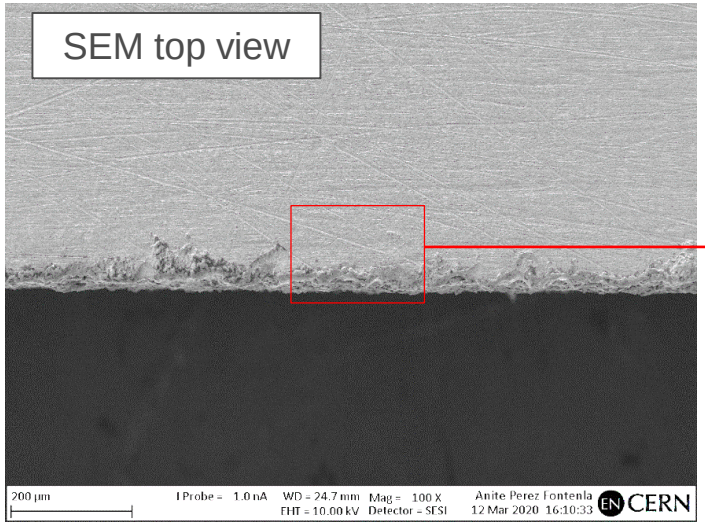
Additional study on Cu-OFE collimator

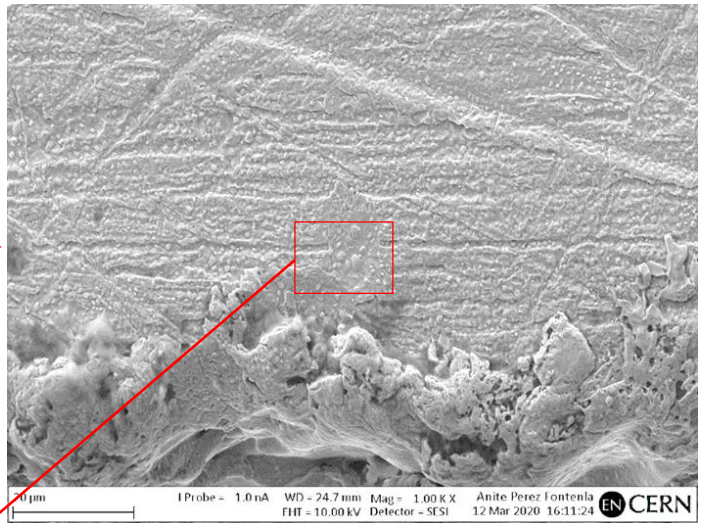


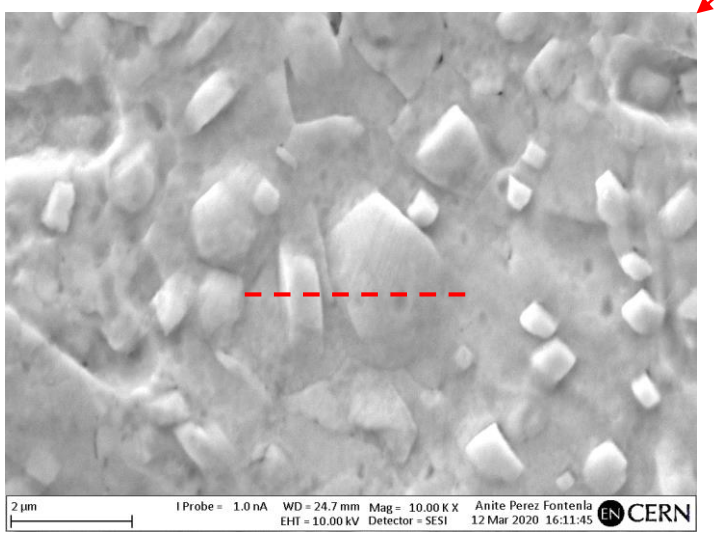
Removed volume: 10 x 8 x 5 µm^3

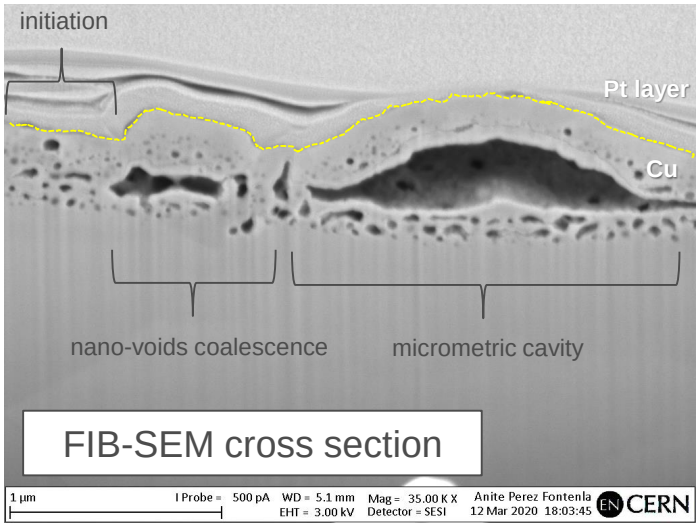
Superficial layer of Cu (at ≈ 0.5 µm depth from the surface) heavily affected by pores

Similar features are reported in the bibliography due to penetration of relatively insoluble gases (hydrogen)









Proposed studies

- A list of candidate materials was carefully defined as alternative to Cu-OFE for manufacturing of a future RFQ with better performance

Cu-OFE	$CuCr_1Zr$	$Cu_{98}Be_2$	Nb	Ta	Ti_6Al_4V
Forged and annealed (current base material)	Forged, solution annealed and aged	Hard temper (precipitation hardening)	RF purity (RRR300)	Melted quality Annealed	Premium grade β-forged

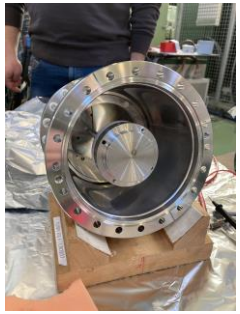
- The materials were selected based on:
  - Their usability for meter-long high gradient RF cavities and,
  - Their potential resistance to **blistering** and breakdown phenomena;

Relevant testing parameters
- Radiation dose
- Ion energy
- Angle of incidence
- Temperature of the “target”

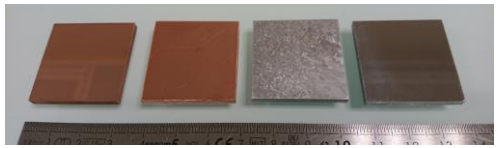
Relevant metallurgical aspects
- Purity
- Grain size
- Pre-existing defects
- Mechanical properties
- Crystal orientation at surface

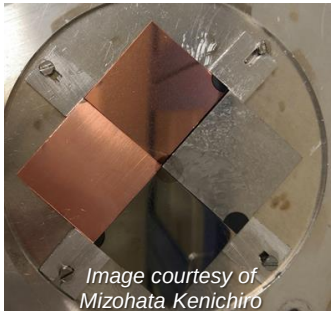
Proposed studies

- Combined H^- source test stand + pulsed DC:
  - Particle type H^- ions at 45 keV
  - Different incidence angles are possible
  - Electrodes of Ø80 mm and Ø60 mm



- Helsinki's system:
  - Particle type H_2^+ at 90 keV
  - Different incidence angles are possible
  - Sample holder ~ Ø100 mm → simultaneously 4 samples (30 mm x 30 mm)

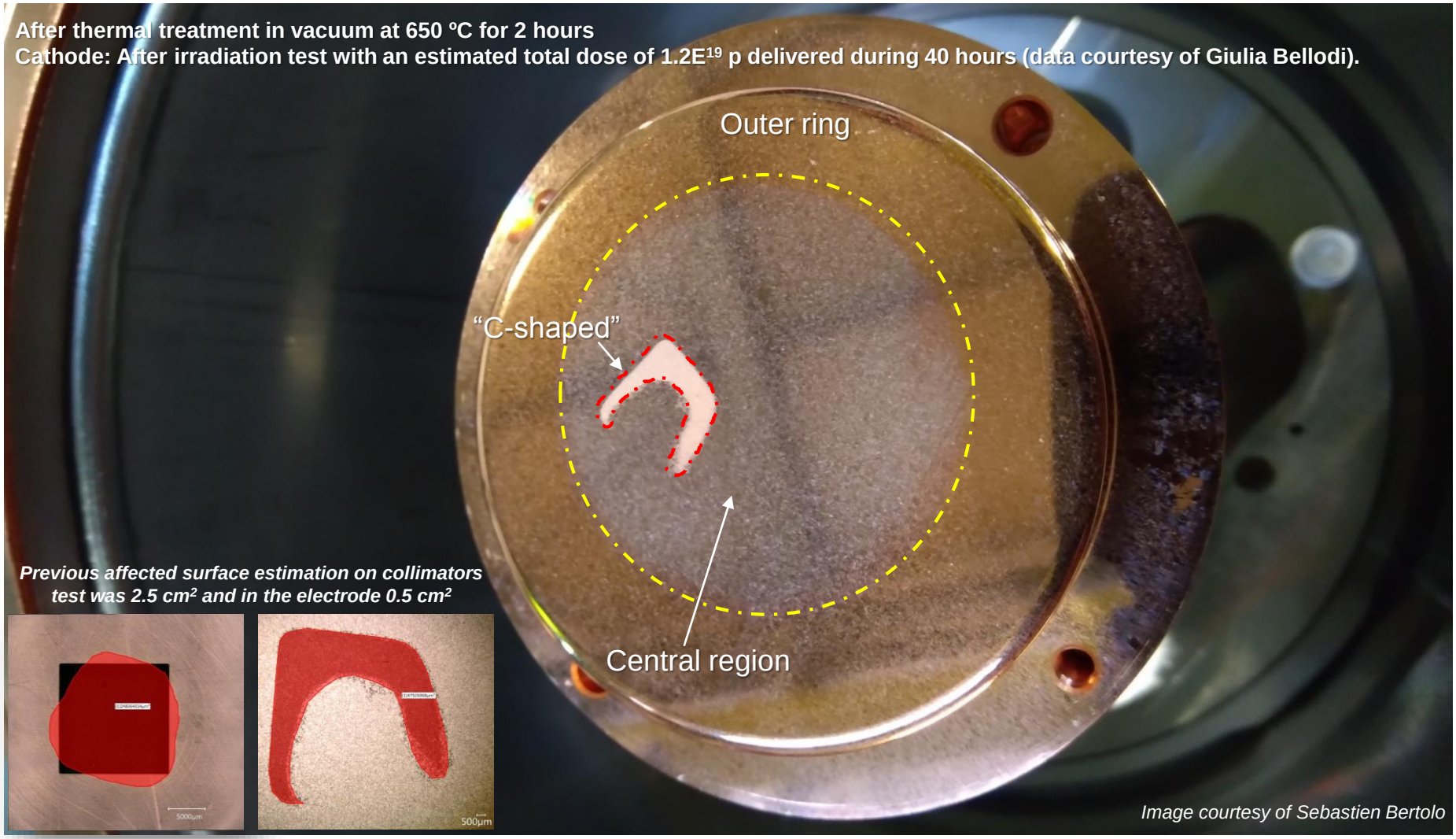




		Combined H^- source + DC testing at CERN						Helsinki's system					
		Cu-OFE	$CuCr_1Zr$	$Cu_{98}Be_2$	Nb	Ta	Ti_6Al_4V	Cu-OFE	$CuCr_1Zr$	$Cu_{98}Be_2$	Nb	Ta	Ti_6Al_4V
~ Dose 1.0 x 10^{19} p/cm^2	1												
	4	n. a.											

Tested *Manufacturing completed* *Material purchased*

Results: Cu-OFE electrode

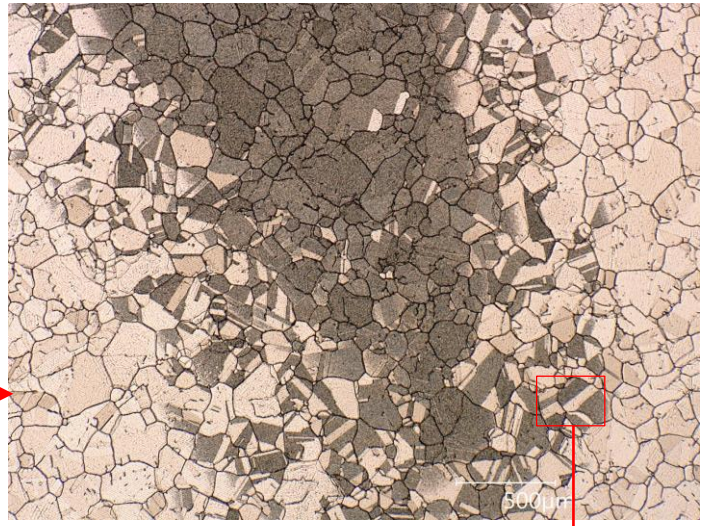


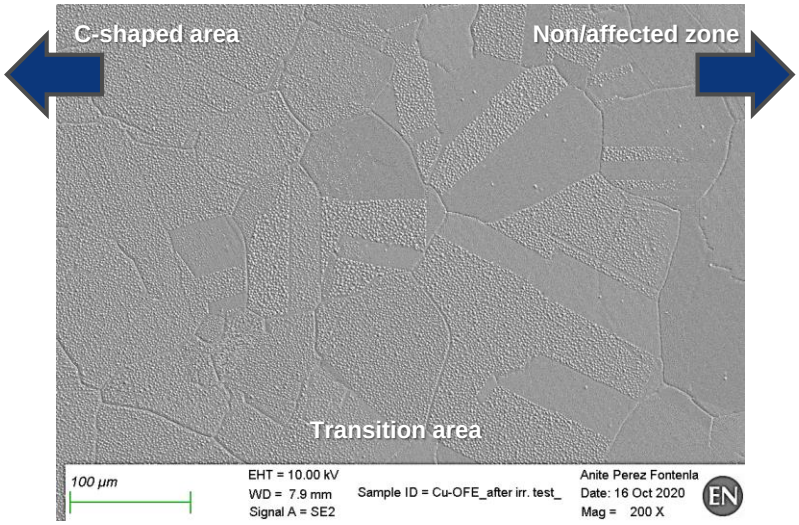
Results: Cu-OFE electrode

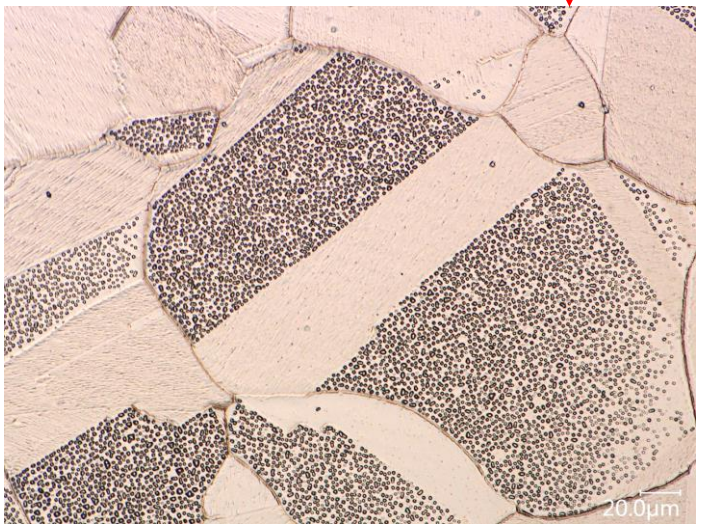
In the transition from irradiated spot to non-irradiated zones, some grains are more affected than others

All the C-shaped area presents blisters

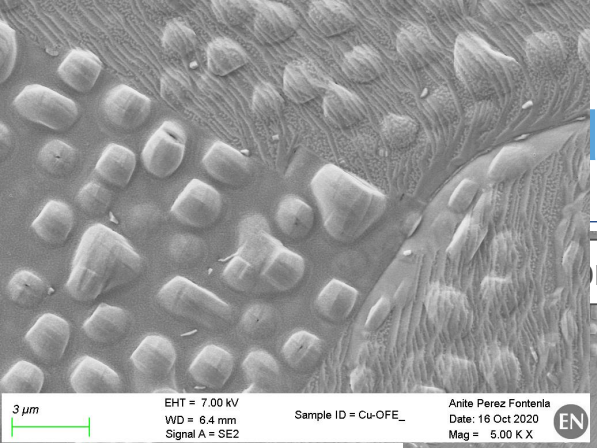
Why C-shaped? Investigations are on-going



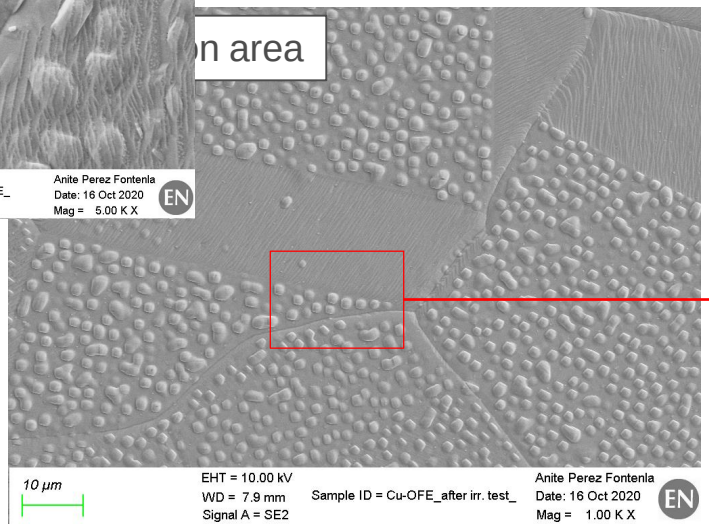


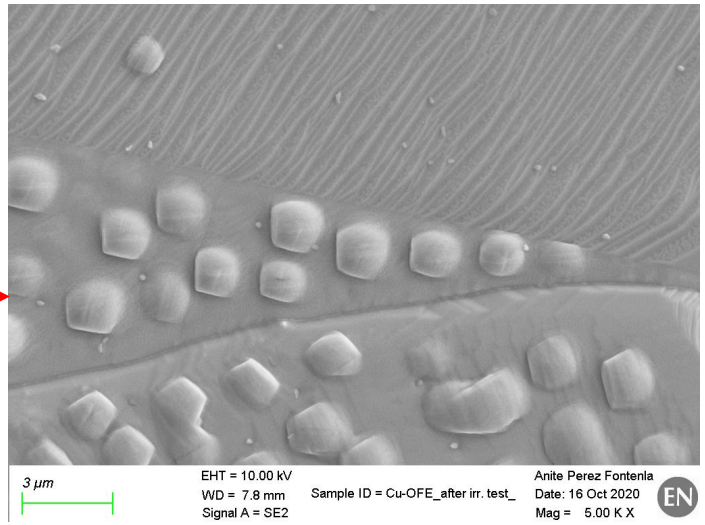


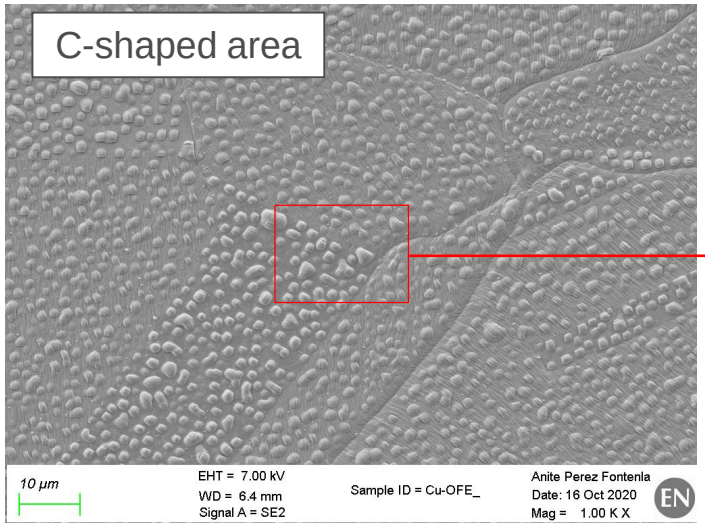
-OFE electrode

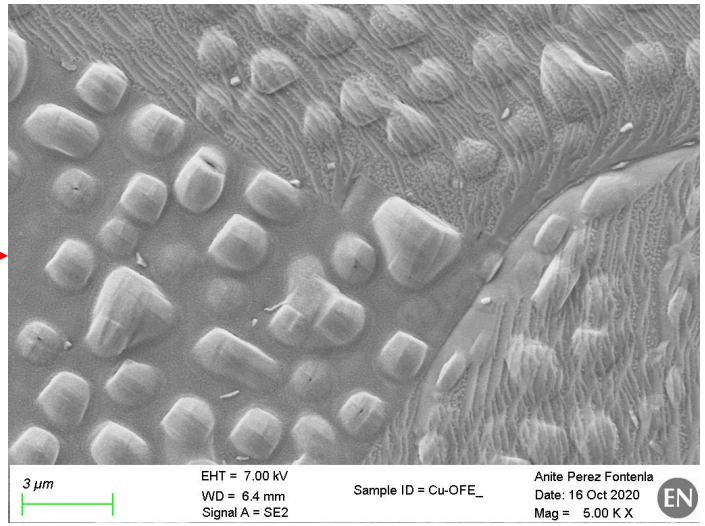


The blisters present small variations in size diameter and morphology depending on the grain orientation





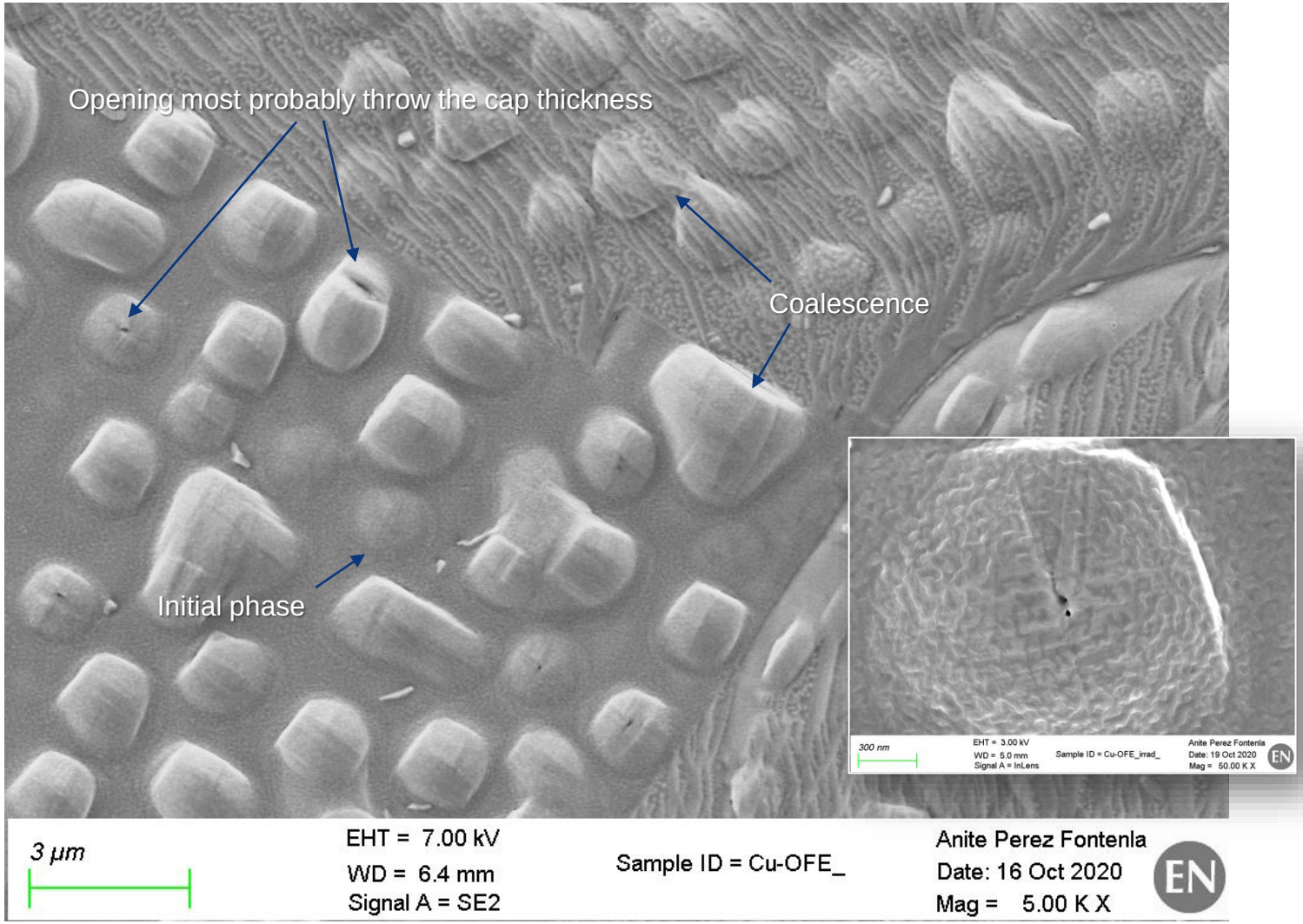




Results: Cu-OFE electrode

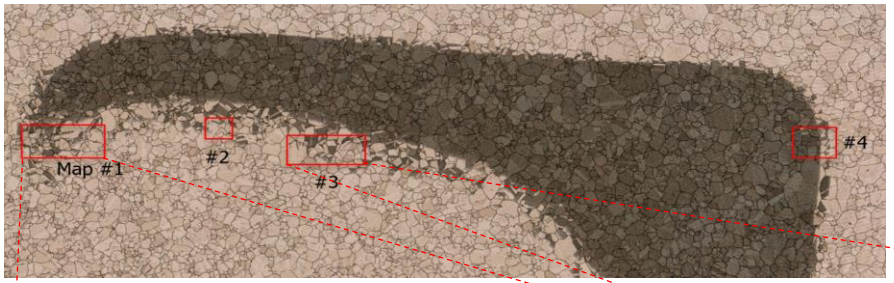
Blisters in different phases of growing are perceptible and in many sites coalescence of two or three blisters is observable

Some of the blisters present openings (holes)



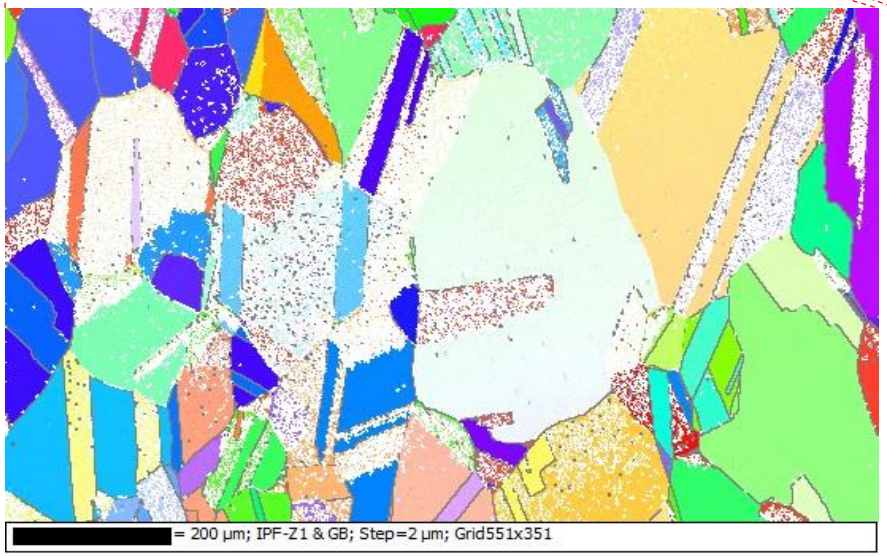
Results: Cu-OFE electrode

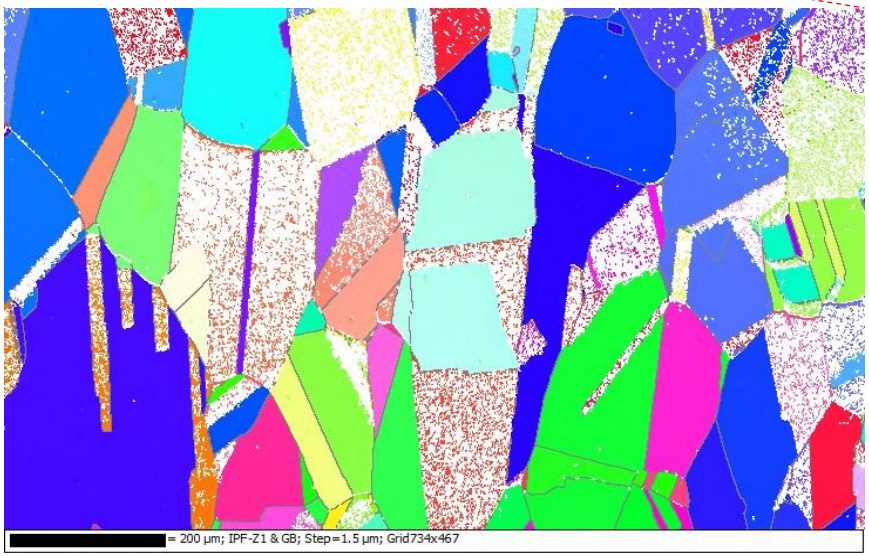
The relation between the blisters location and crystallographic orientation was evaluated experimentally using the non-destructive technique Electron Back-Scattered Diffraction (EBSD) into the SEM.



Using the common color-codes for cubic materials, it can be observed that in general the blue and green grains (corresponding to crystal directions [111] and [101] respectively) were less affected by blisters than the red grains (corresponding to crystal direction [001])

Inverse Pole Figures coloring maps in Z direction (IPF-Z)





Results: Cu-OFE specimen (Helsinki)

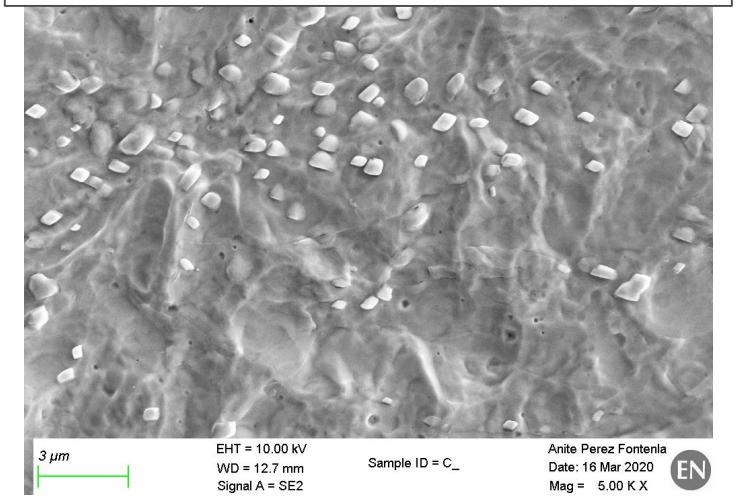
Presence of blisters randomly distributed on the Cu-OFE surface was confirmed with both used doses in Helsinki (is not possible to correlate with grain orientations at the transition areas due to the surface roughness)

Size, density and shape of the blisters on the collimator are similar to the ones observed at the lower dose test region

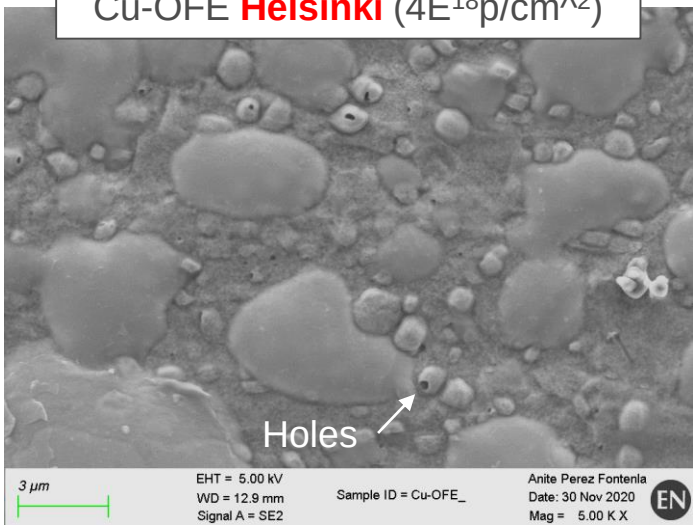
In the case of higher dose, different features appeared (larger blisters?) in addition to the smaller blisters

Some blisters present holes/openings at the cap

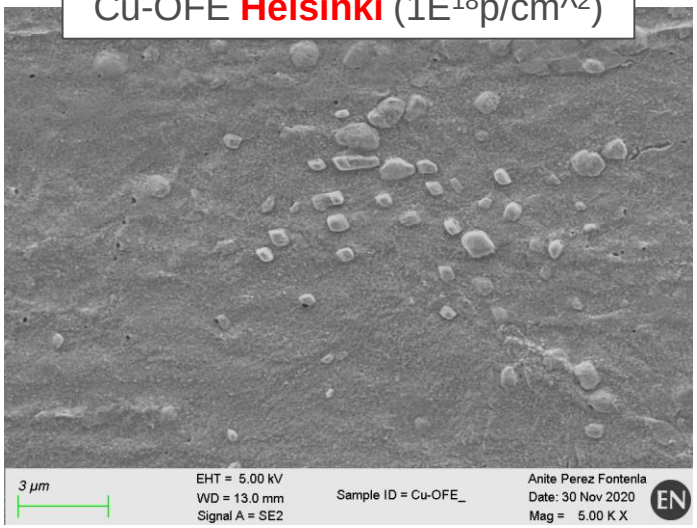
Cu-OFE Collimator **CERN** (1.7E^{19}p/cm^^{2})



Cu-OFE **Helsinki** (4E^{18}p/cm^^{2})

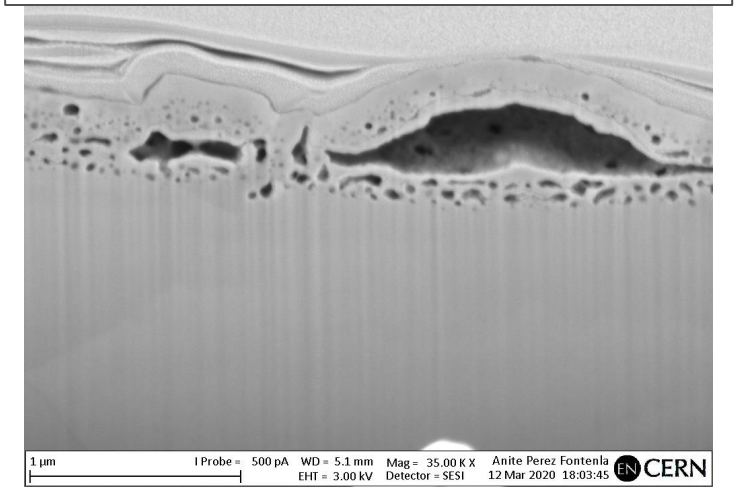


Cu-OFE **Helsinki** (1E^{18}p/cm^^{2})



Results: Cu-OFE specimen (Helsinki)

Cu-OFE Collimator **CERN** (1.7E^{19}p/cm^^{2})



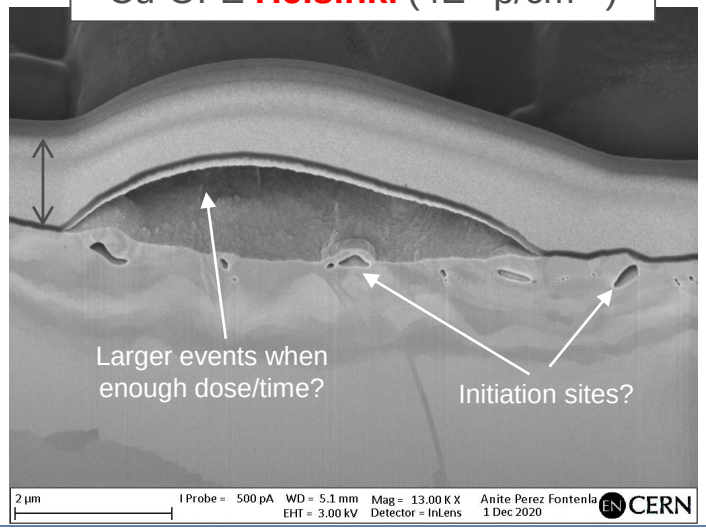
Nanopores observed in the collimator are only visible punctually in the affected layer on the Cu-OFE irradiated samples in Helsinki

The blister cap thickness on the collimator is similar to the lower dose test in Helsinki and similar to some features in the higher dose (initiation/nucleation sites?);

Presence of larger blisters containing smaller events is confirmed and a carbon layer is visible;

Cu-OFE **Helsinki** (4E^{18}p/cm^^{2})

Pt deposit

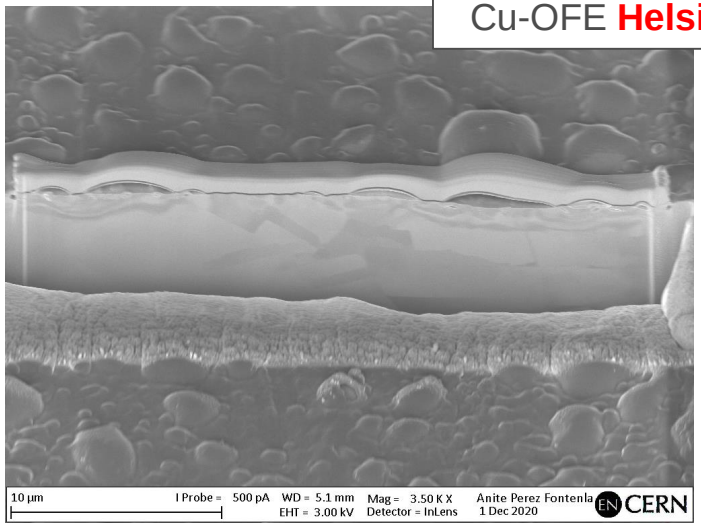


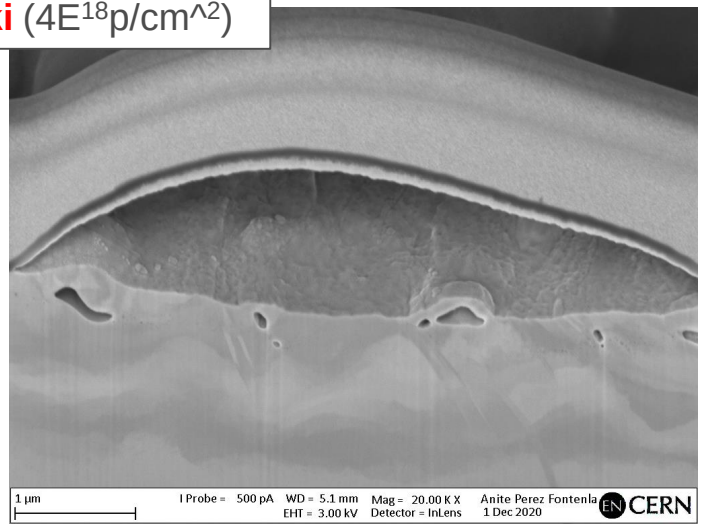
Cu-OFE **Helsinki** (1E^{18}p/cm^^{2})

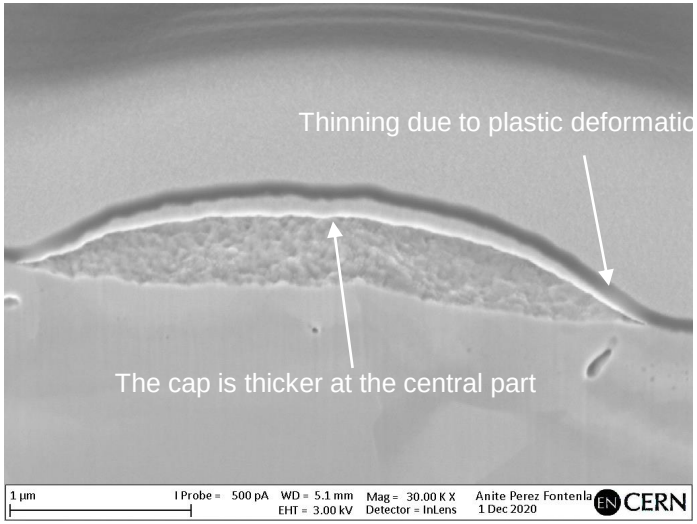
Pt deposit

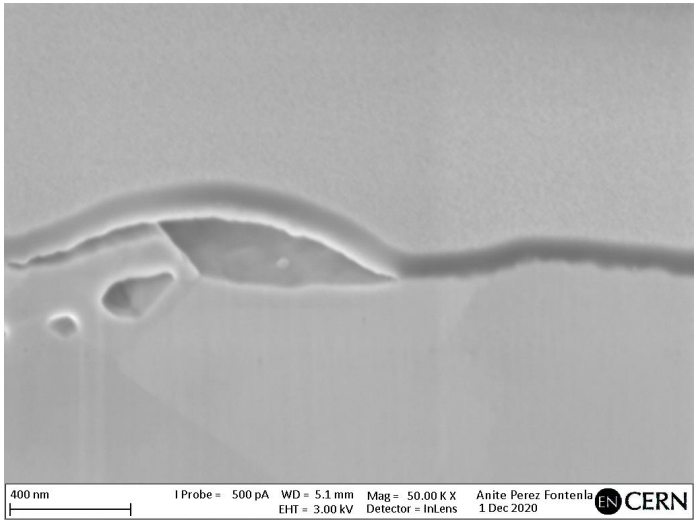
Results: Cu-OFE specimen (Helsinki)

Cu-OFE **Helsinki** (4E^{18}p/cm^^{2})



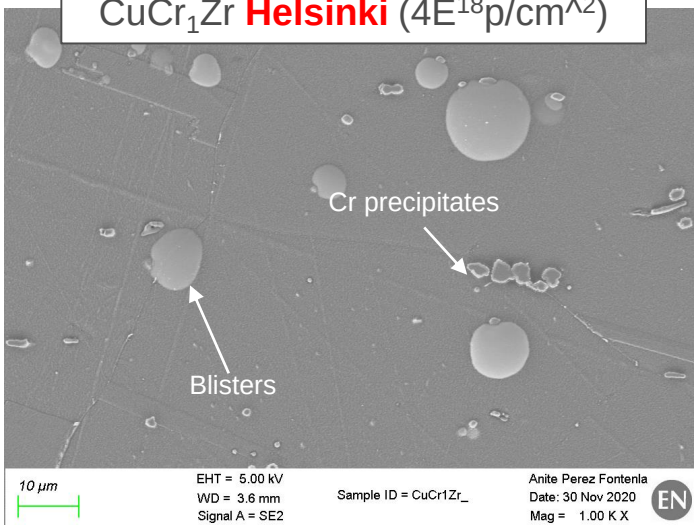




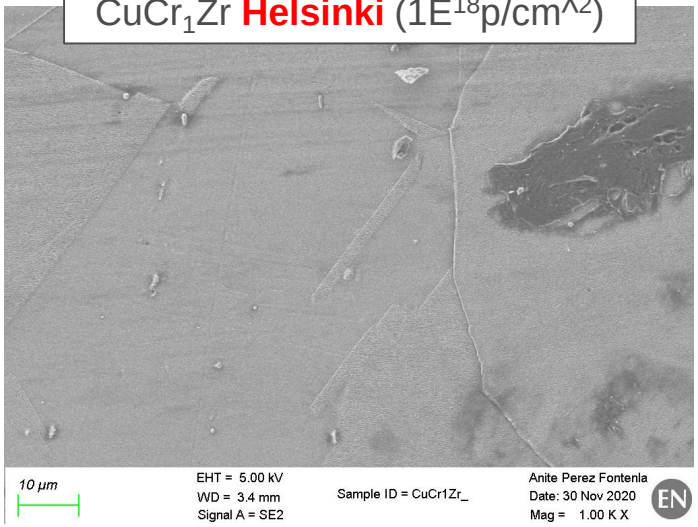


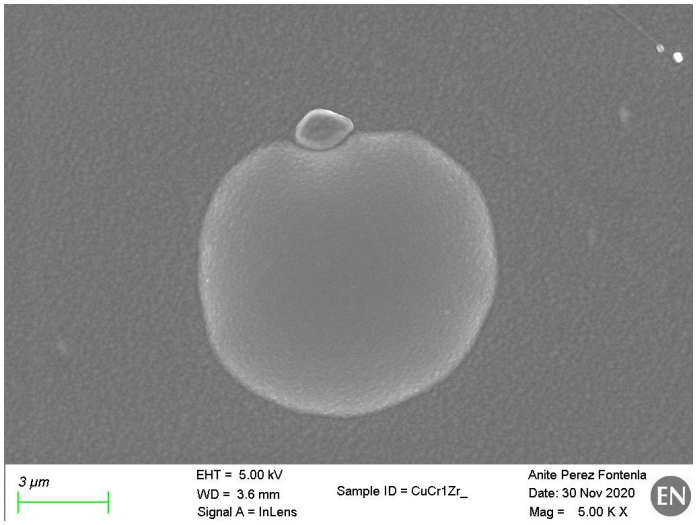
Results: $CuCr_1Zr$ specimen (Helsinki)

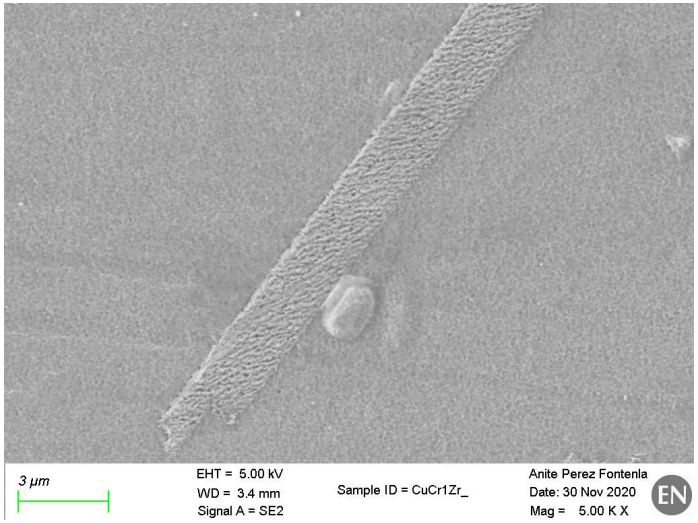
$CuCr_1Zr$ **Helsinki** ($4E^{18}p/cm^{\wedge 2}$)



$CuCr_1Zr$ **Helsinki** ($1E^{18}p/cm^{\wedge 2}$)

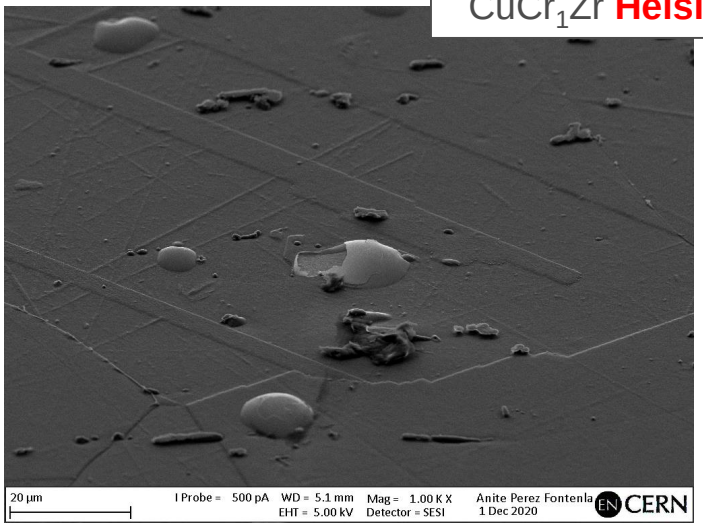


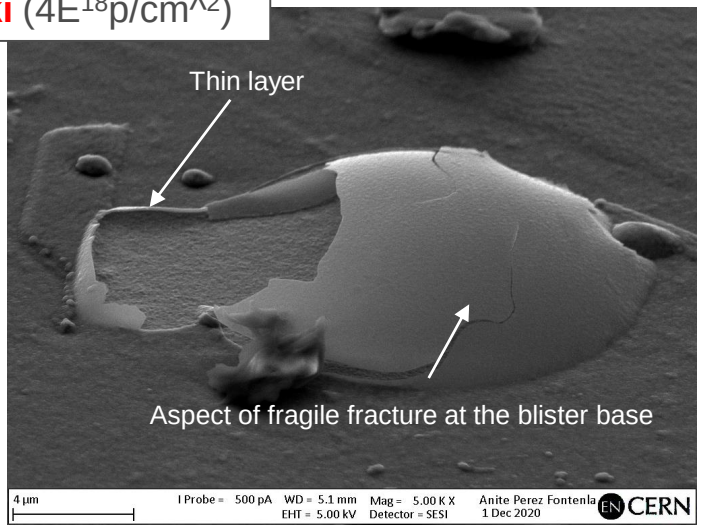


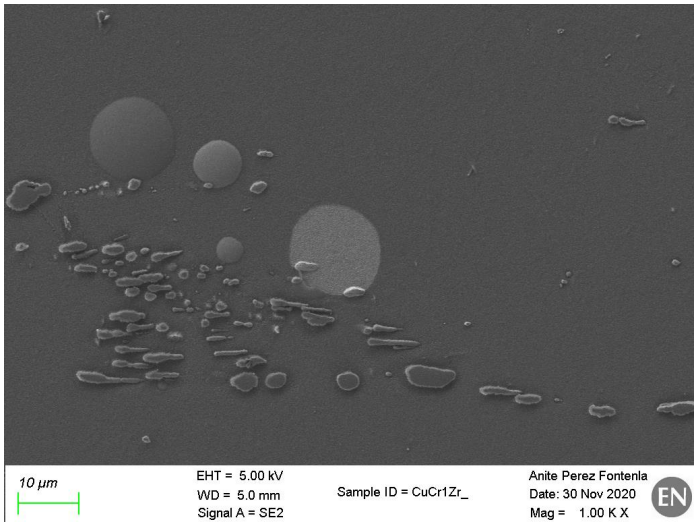


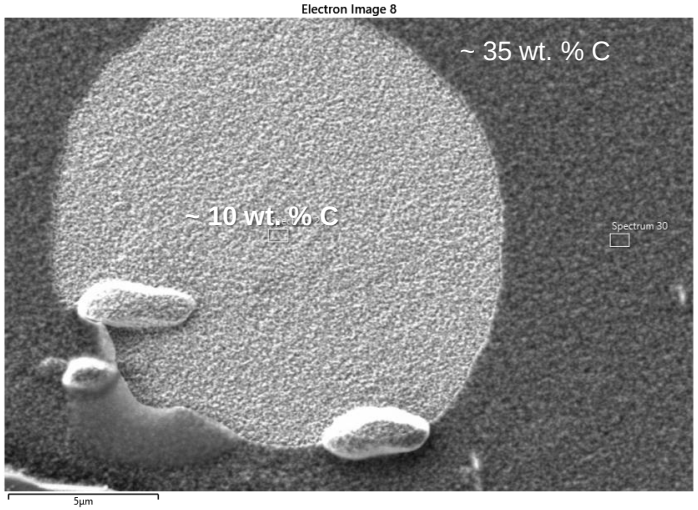
Results: $CuCr_1Zr$ specimen (Helsinki)

$CuCr_1Zr$ **Helsinki** ($4E^{18}p/cm^{\wedge 2}$)



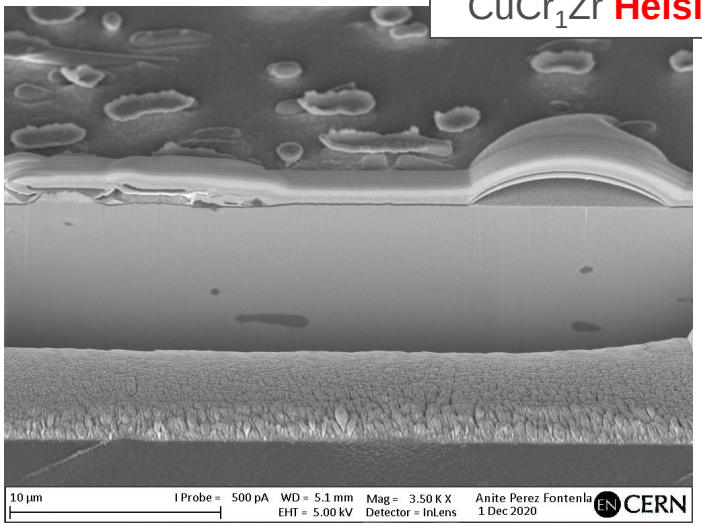


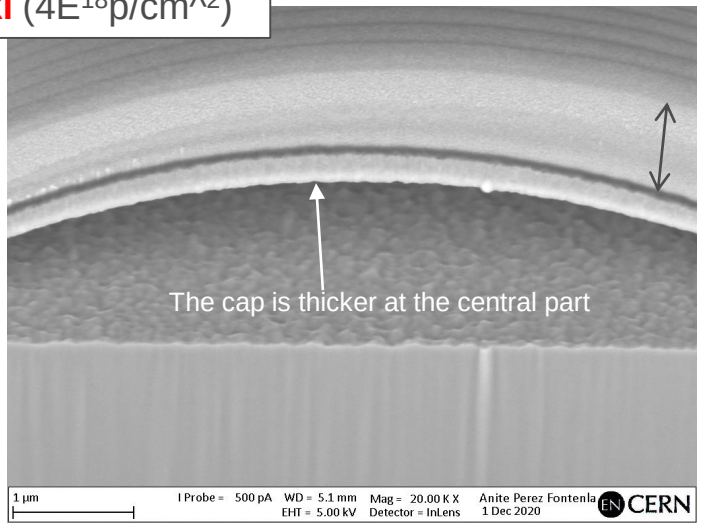


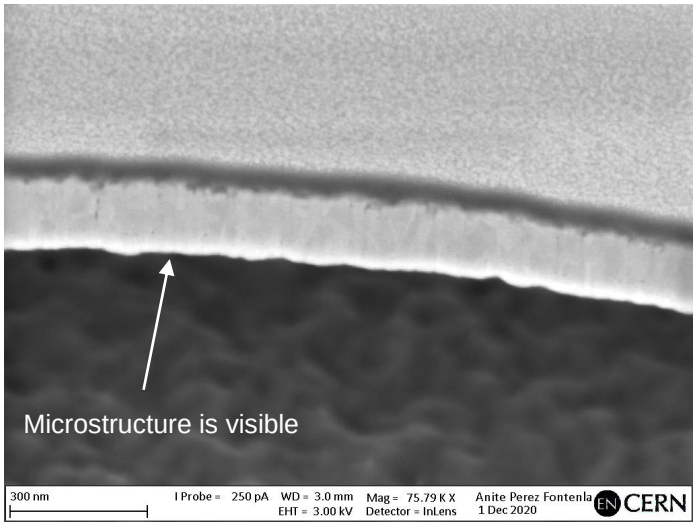


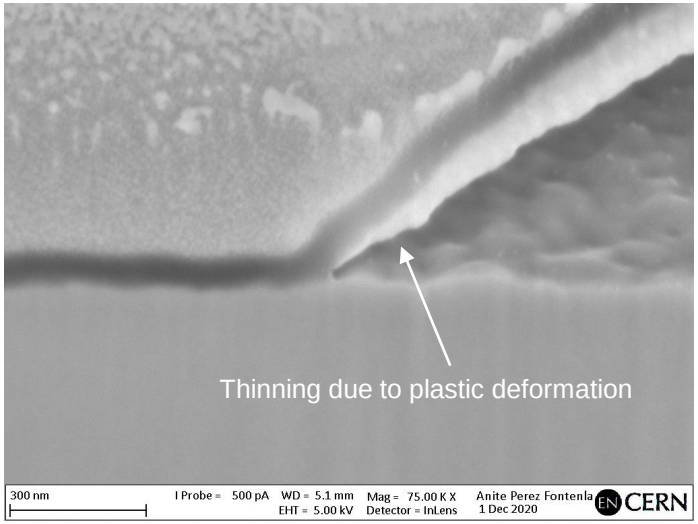
Results: $CuCr_1Zr$ specimen (Helsinki)

$CuCr_1Zr$ **Helsinki** ($4E^{18}p/cm^{\wedge 2}$)



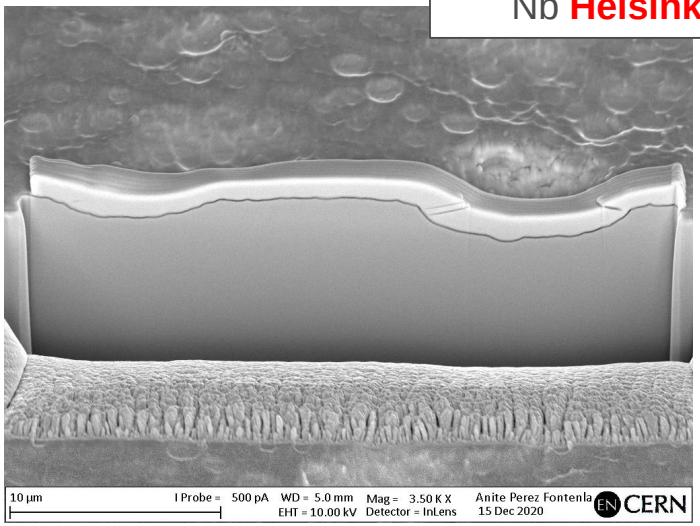


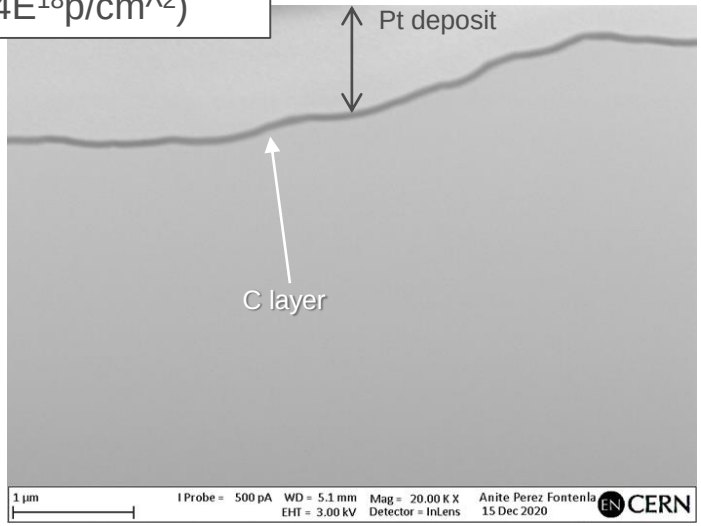




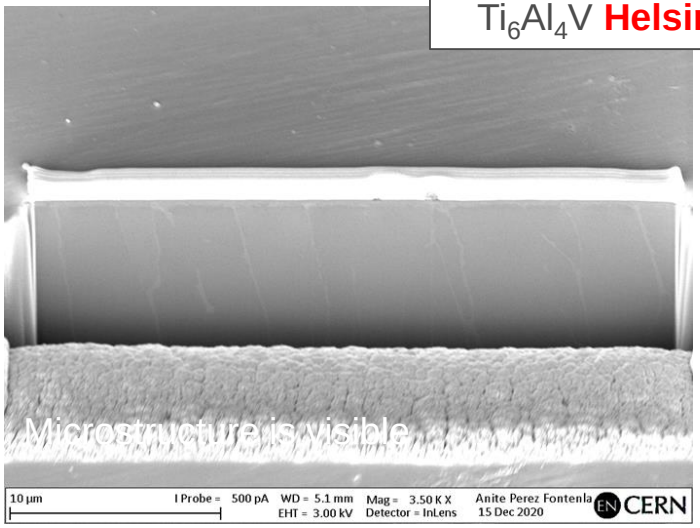
Results: Nb and Ti_6Al_4V (Helsinki)

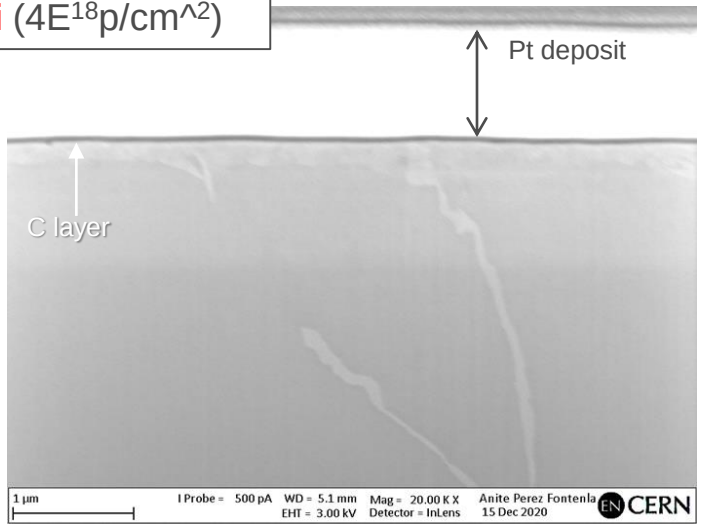
Nb **Helsinki** (4E^{18}p/cm^^{2})





Ti_6Al_4V **Helsinki** (4E^{18}p/cm^^{2})





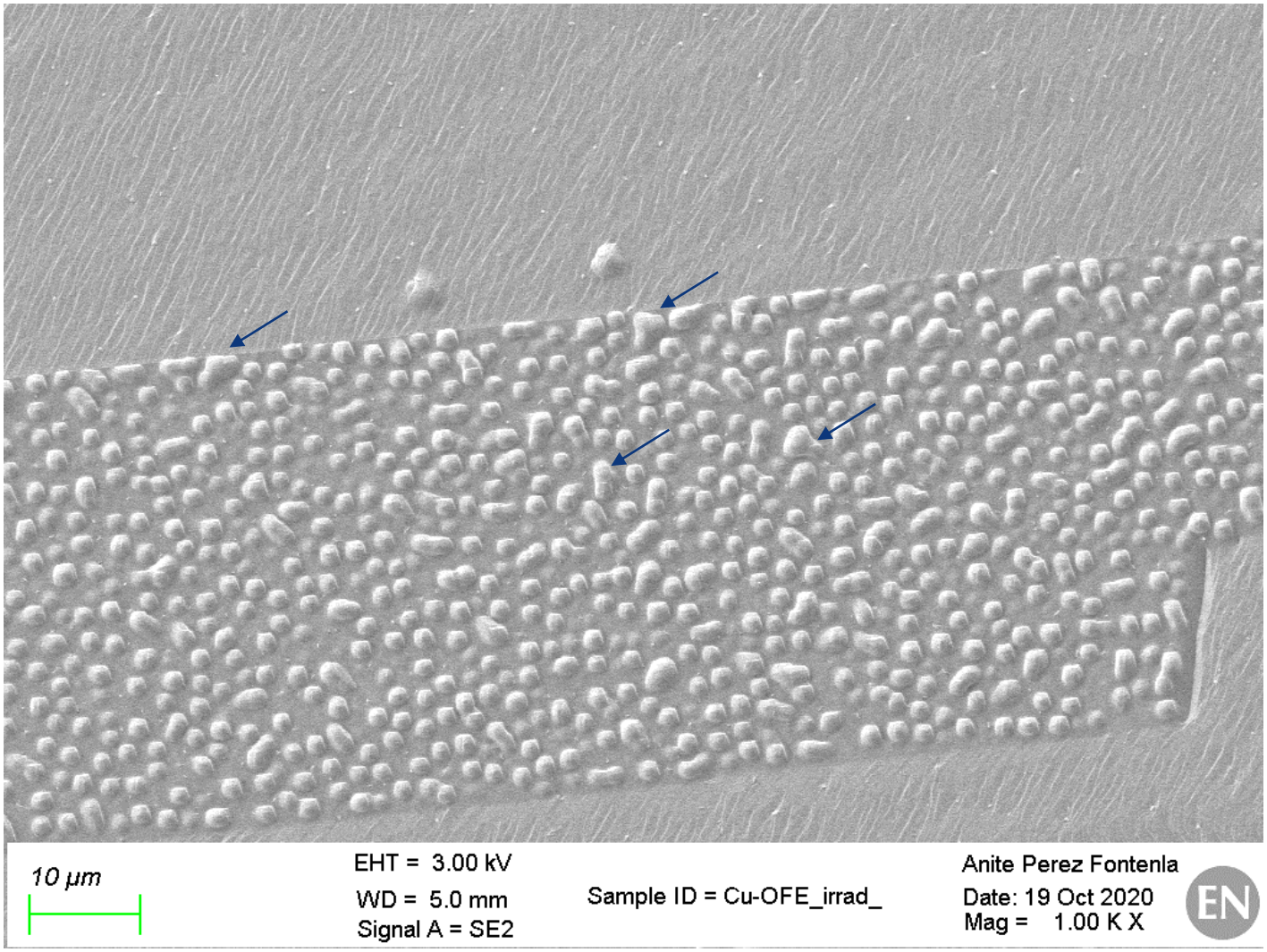
Summary

- The in-situ test in Linac4 RFQ by optical means pointed out the presence of local damage on the Cu surface (BKD craters and “worm-like features”);
- Further investigations with collimators/masks confirmed the presence of blisters induced by the interaction with the beam;
- The size of those events differs but they could be connected → a deeper look to better understand the blister-BKD location relation is needed;
- Combined testing (irradiation + pulsed DC system) was performed on RFQ base material (Cu-OFE). The presence of blister was confirmed and their aspect and location were characterized by SEM and EBSD;
- In addition, other candidate materials for RFQ production were identified and are being studied in similar way in order to compare their performance and resistance to blistering and BKD phenomena;
- The cost and leading time to fabricate and test at CERN large electrodes oriented us to look for testing alternatives and the preliminary results obtained with Helsinki’s system compared well with CERN studies in collimators/masks;
- This flexible and reproducible system allowed us to test simultaneously Cu-OFE, $CuCr_1Zr$, Nb and Ti_6Al_4V specimens with two different doses. Nevertheless, some questions raised up (C layer, bigger blisters at higher doses/time, surface preparation…) → more tests needs to be performed;
- So far, both cuprous materials tested presented blisters (in the case of the CuCr1Zr only at higher dose or on deformed areas) and Nb and Ti_6Al_4V were free of features even at higher doses;
- It’s being a continuous learning process and it is important to mention the implication of a wide range of experts at CERN and Helsinki to evaluate the findings until now and the project support to hire a PhD student.

Thank you for your attention!

CERN | EN ENGINEERING DEPARTMENT

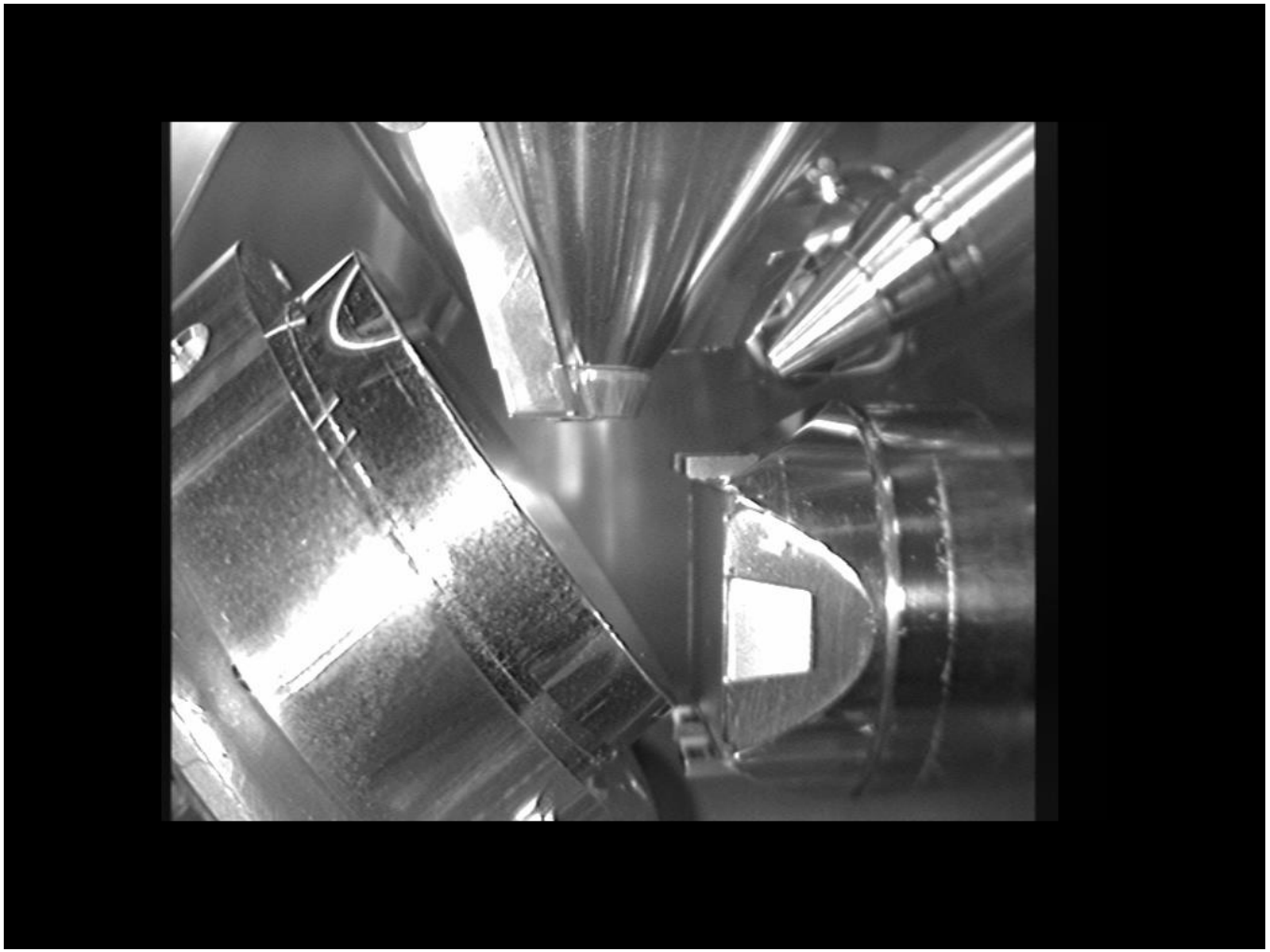
Extra slides



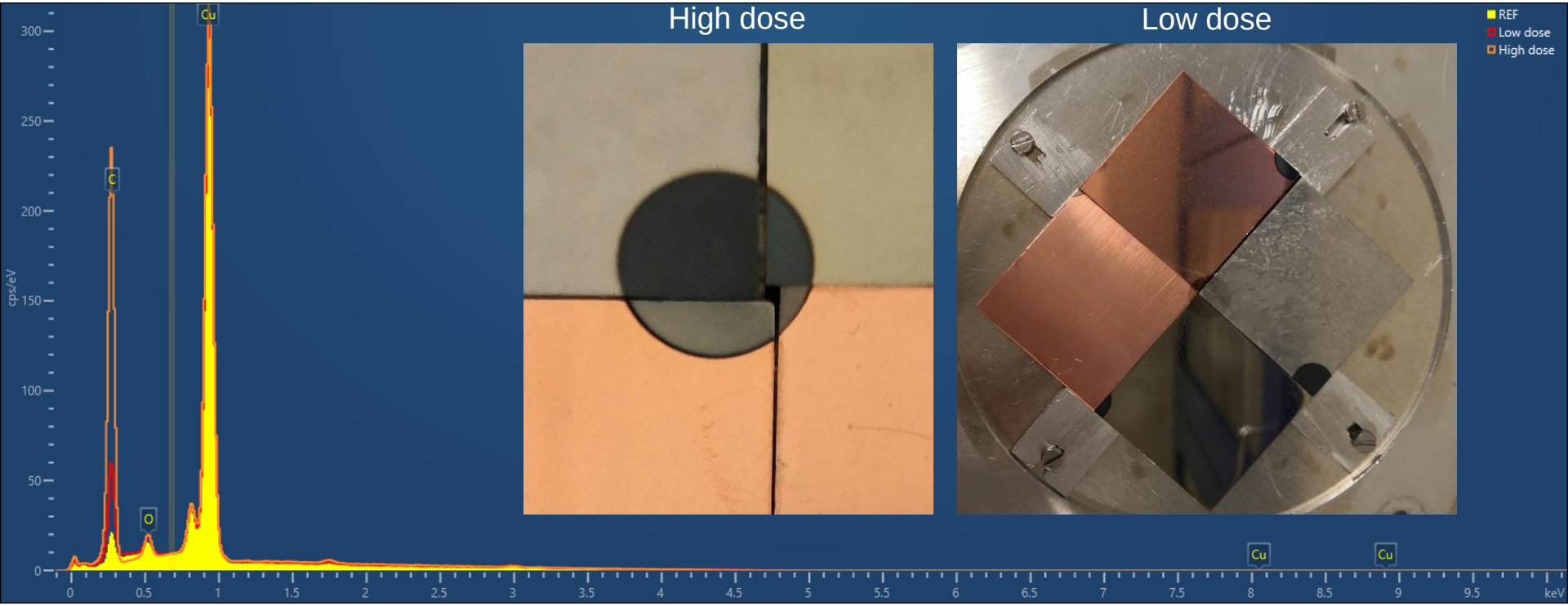
Extra slides

EDS comparison at the central region (SOI-5) and external region (SOI-7)

Extra slides

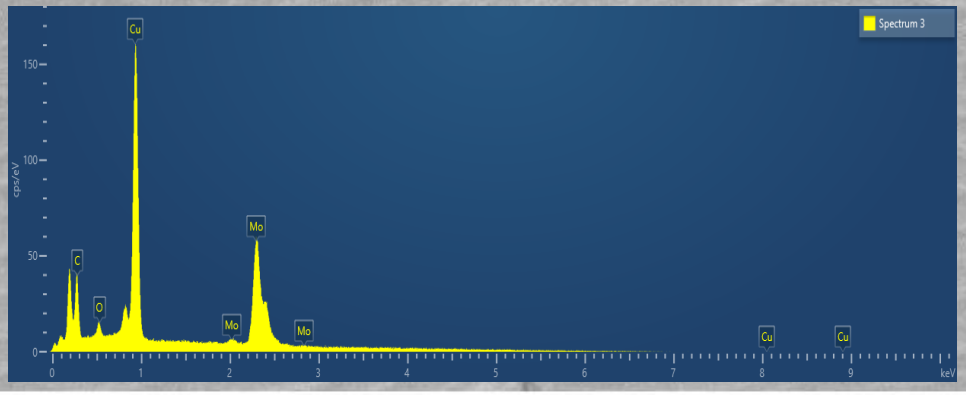


Extra slides



Faceting on the Cu surface is visible independently of the region.
That is a consequence of the thermal treatment performed on the electrode before testing.
Faceting is related with selective evaporation of atoms in non–energetically favourable crystal position thus the appearance varies depending on the grain orientation

Mo rich inclusions are visible all over the Cu surface



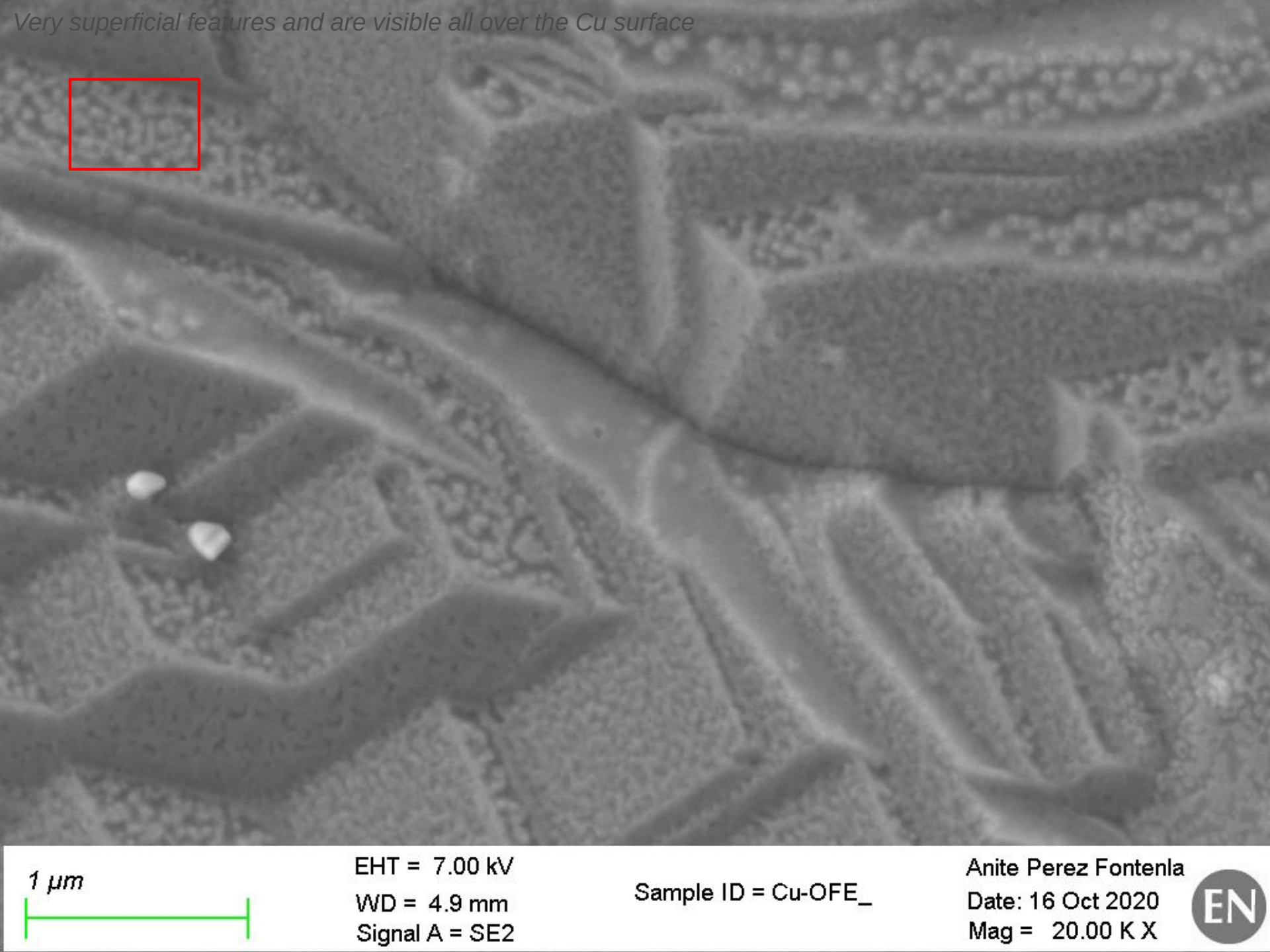
3 µm

EHT = 10.00 kV
WD = 7.9 mm
Signal A = SE2

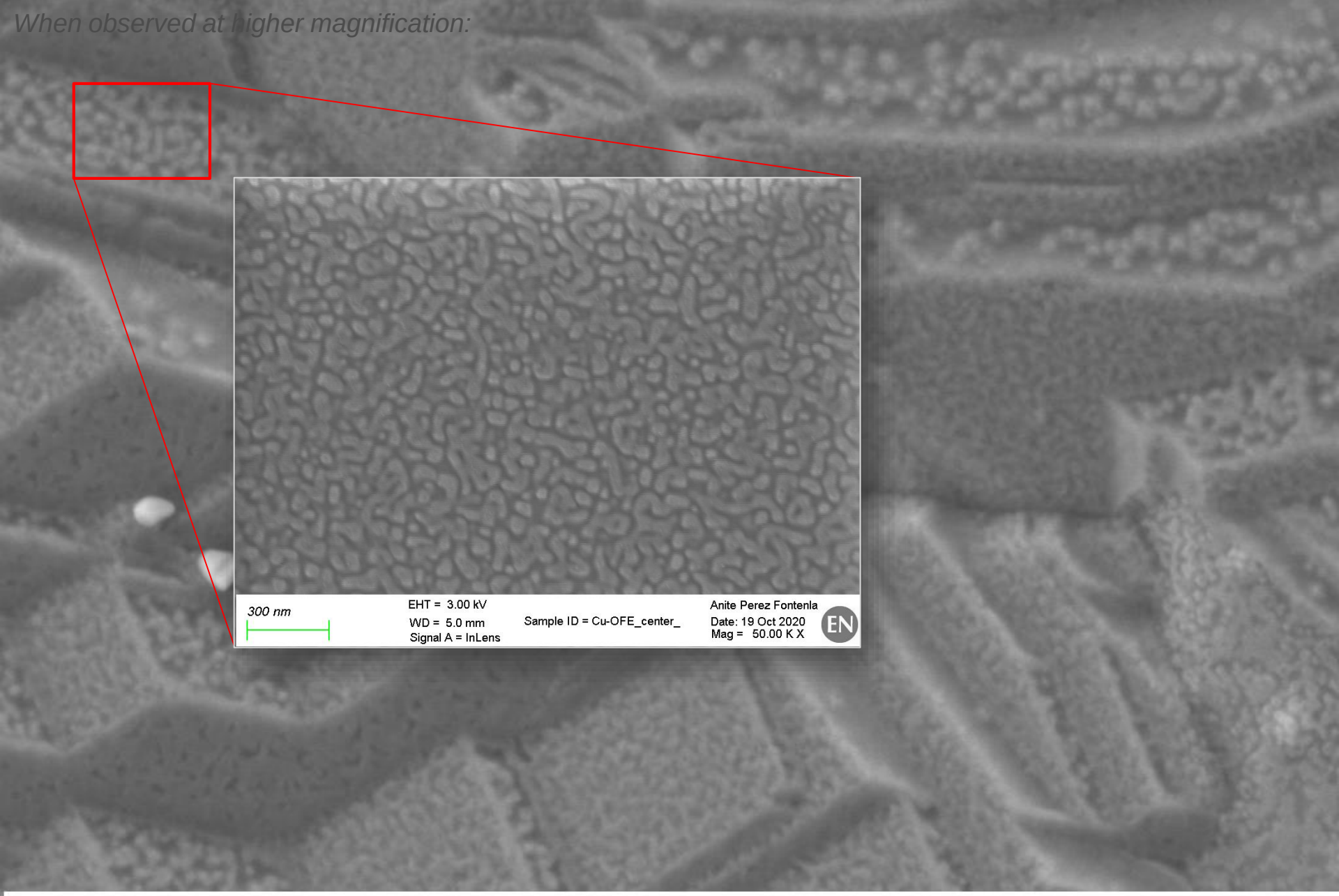
Sample ID = Cu-OFE_after irr. test_

Anite Perez Fontenla
Date: 16 Oct 2020
Mag = 5.00 K X

EN


Very superficial features and are visible all over the Cu surface
1 µm
EHT = 7.00 kV
WD = 4.9 mm
Signal A = SE2
Sample ID = Cu-OFE_
Anite Perez Fontenla
Date: 16 Oct 2020
Mag = 20.00 K X
EN

When observed at higher magnification:



1 µm

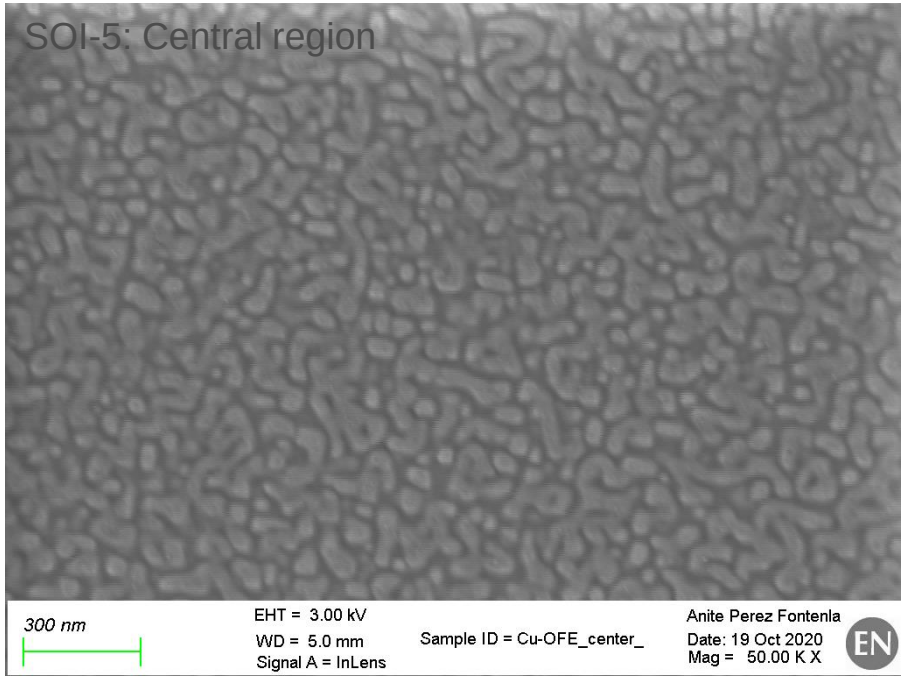
EHT = 7.00 kV
WD = 4.9 mm
Signal A = SE2

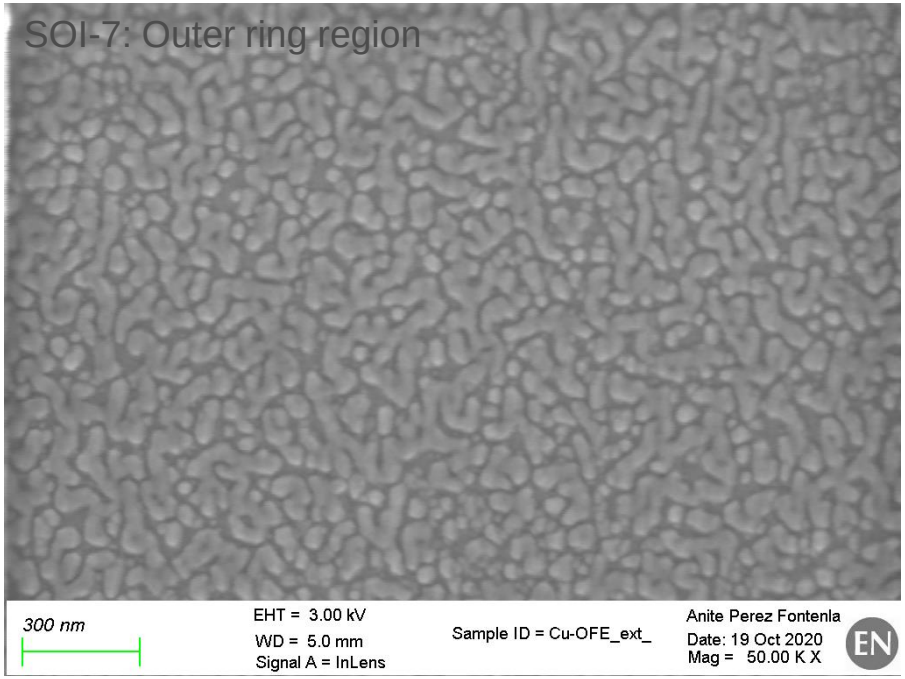
Sample ID = Cu-OFE_

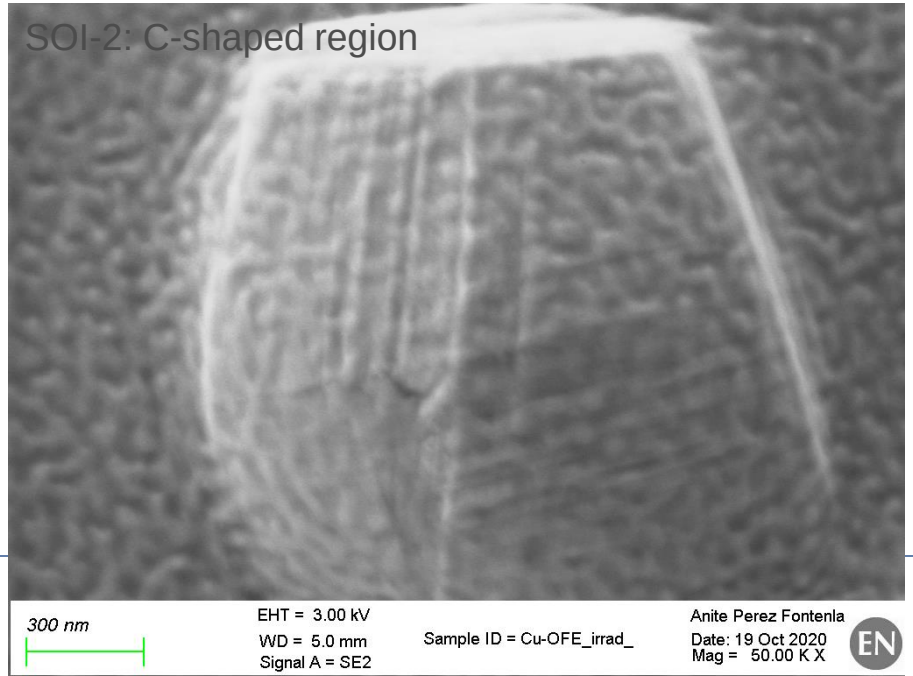
Anite Perez Fontenla
Date: 16 Oct 2020
Mag = 20.00 K X

EN

When observed at higher magnification:

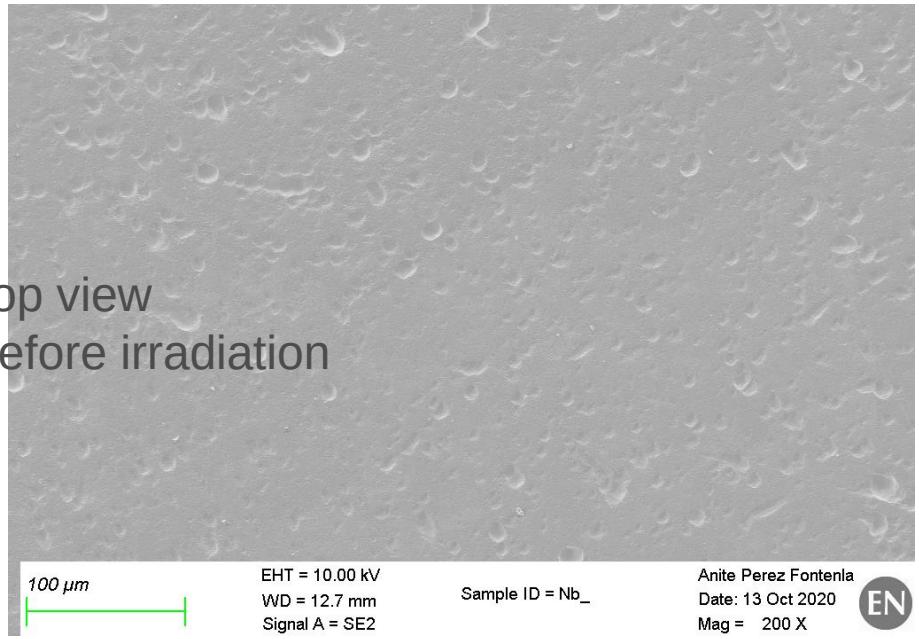


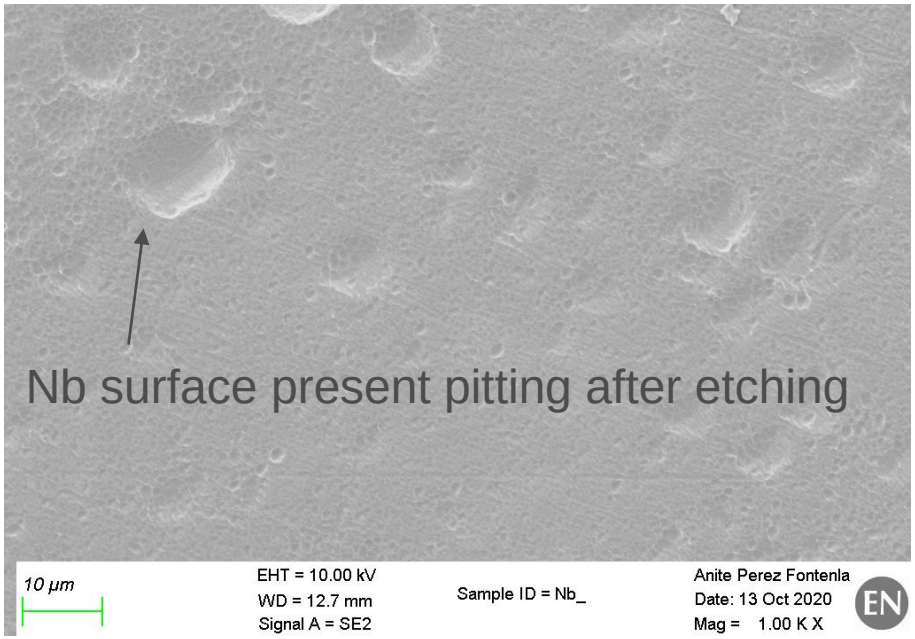




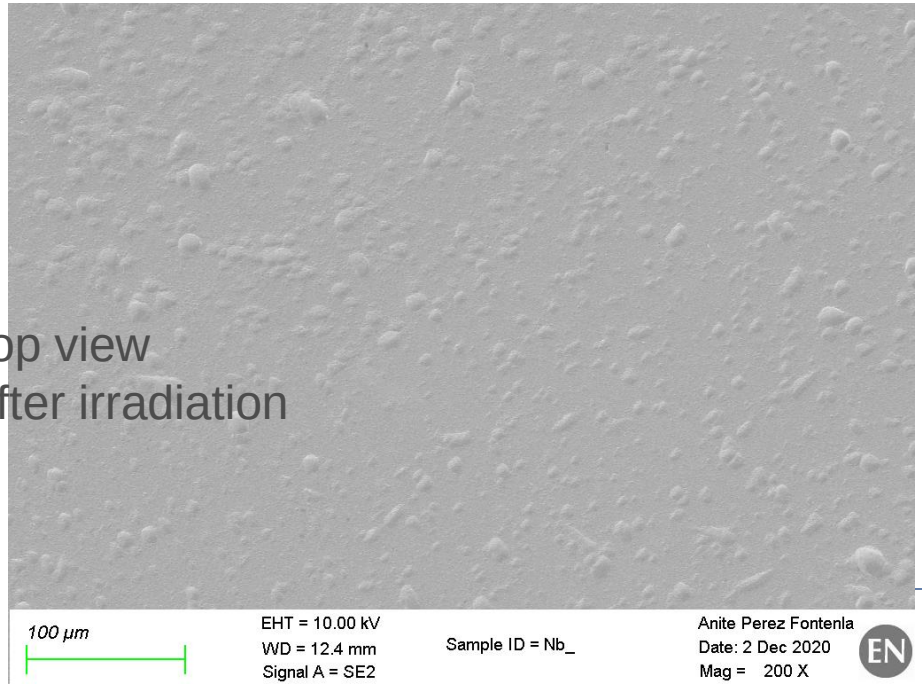
Comparison of Nb sample same location before and after irradiation (dose 4E18 H/cm^2)

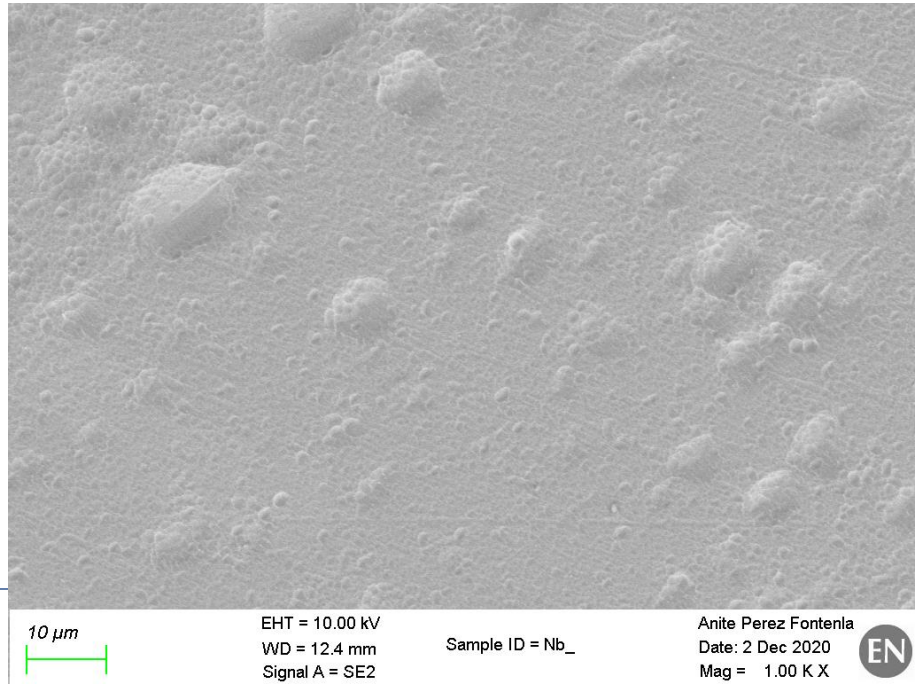
Top view
Before irradiation





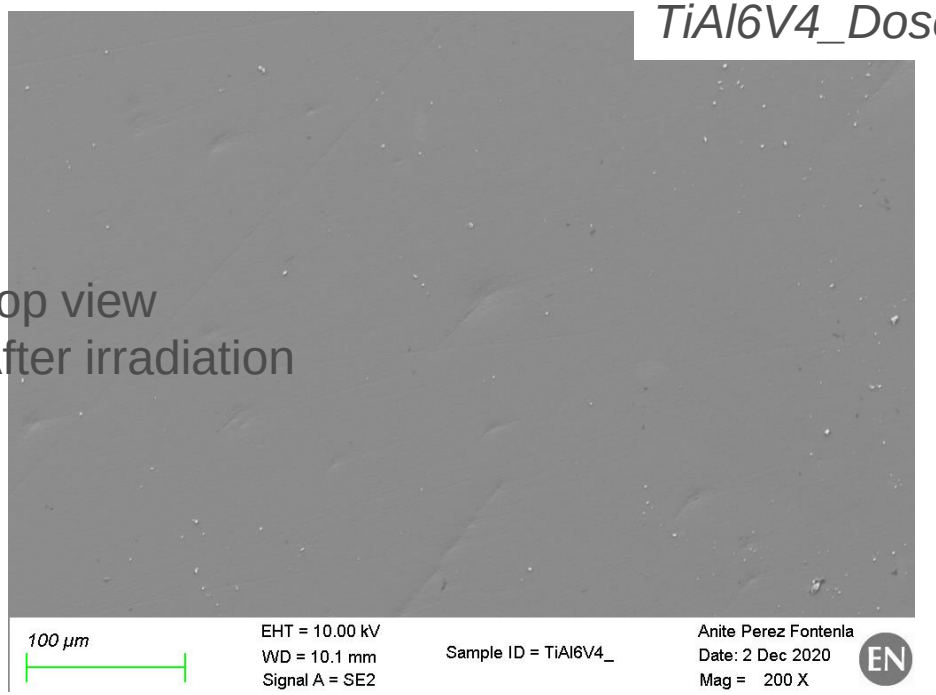
Top view
After irradiation

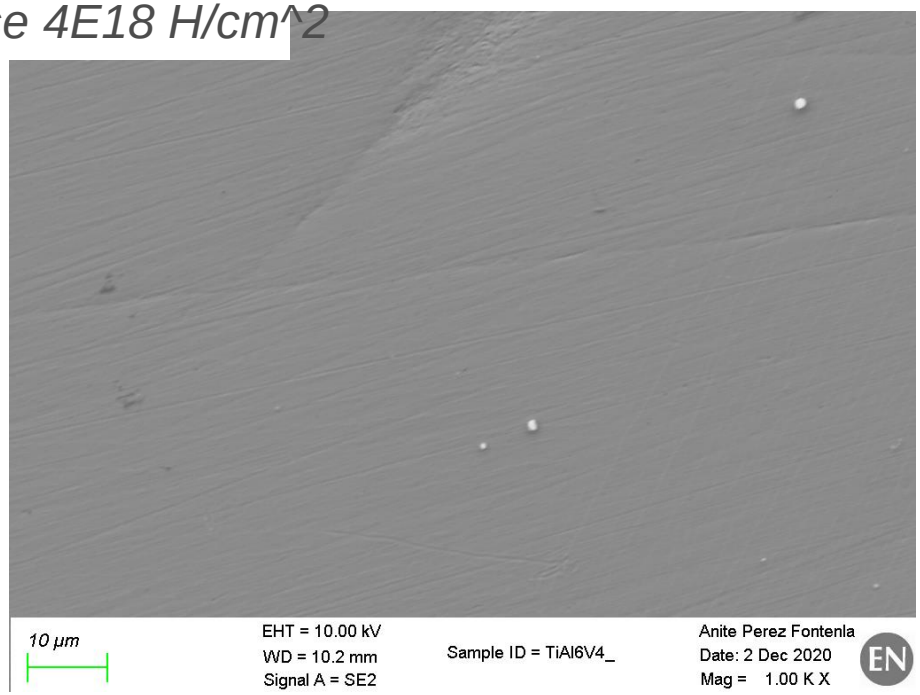




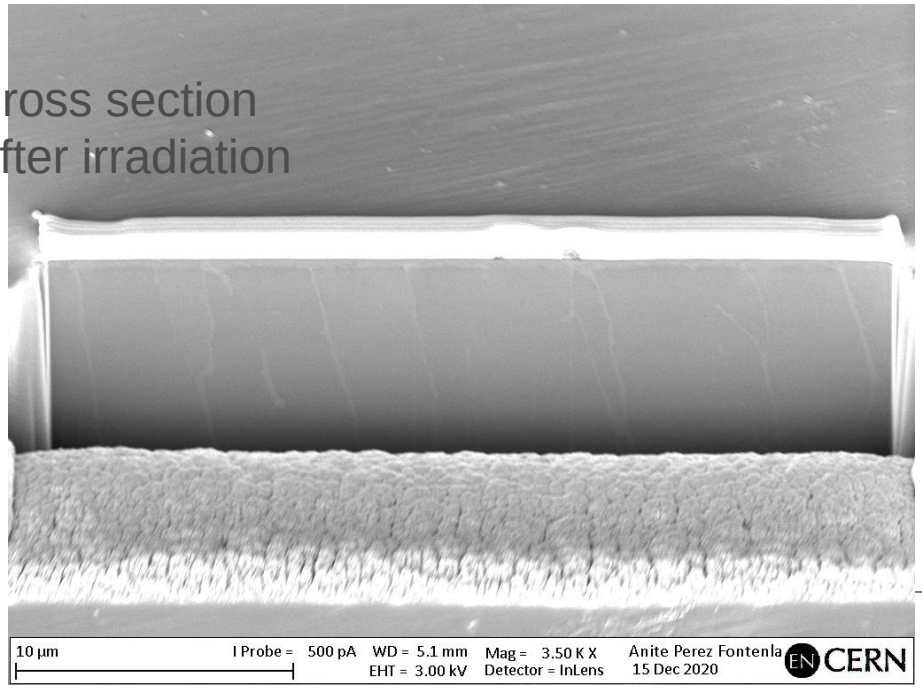
TiAl6V4_Dose 4E18 H/cm^2

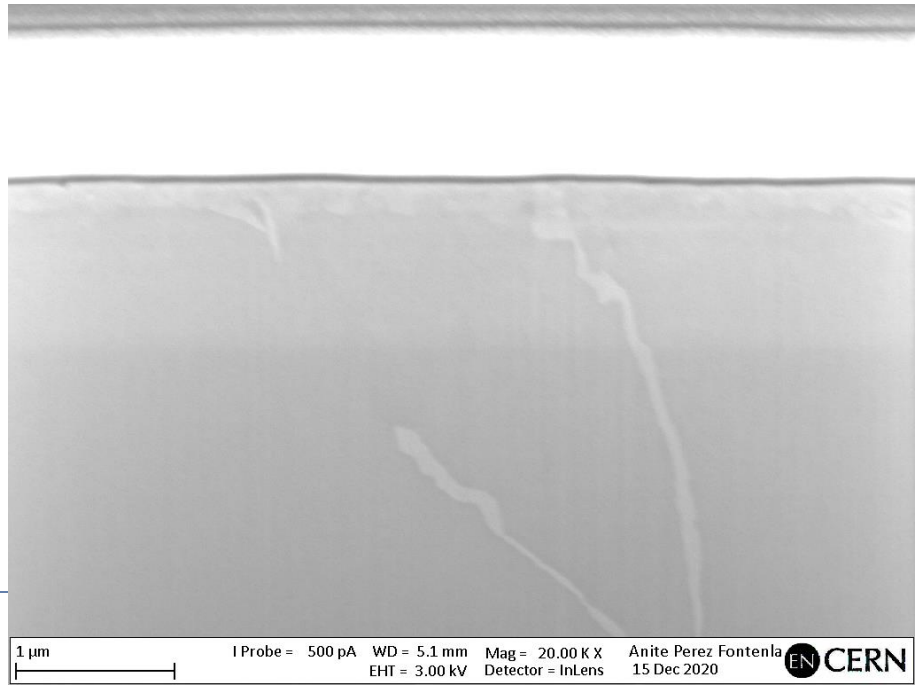
Top view
After irradiation





Cross section
After irradiation





Extra slides

Lowers the cohesive strength of interfaces (e.g. "cortical layer")