

# MeVArc 2024

Tahoe City, USA

Catarina Serafim

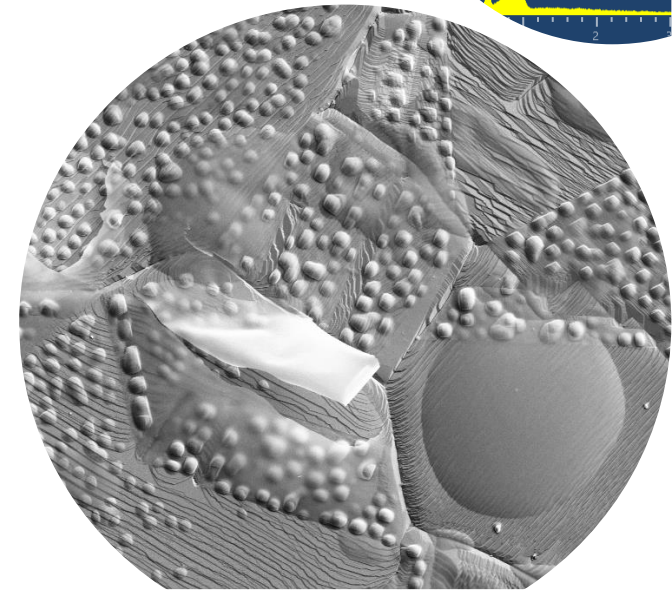
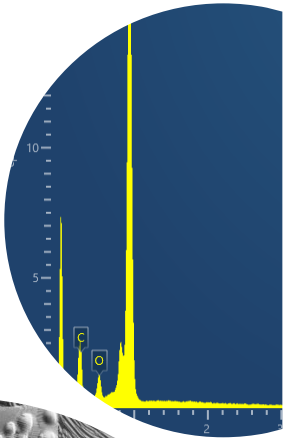
*Study of different materials in high voltage  
breakdown tests in the DC system, before and after  
H- low voltage irradiation*

*Supervisors:*

*Sergio Calatroni, CERN*

*Flyura Djurabekova, University of Helsinki*

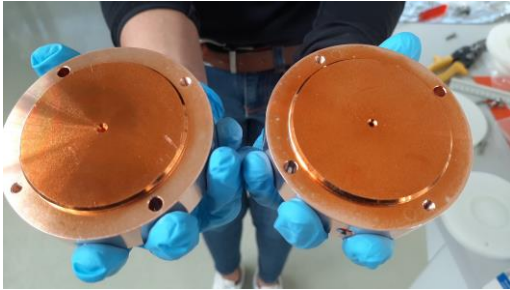
6<sup>th</sup>, March, 2024



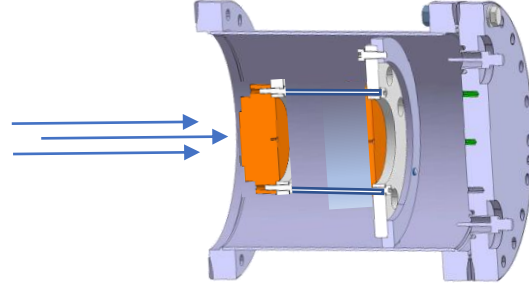
# Procedure

## 1<sup>st</sup> pair of electrodes

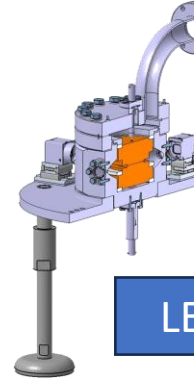
Treatment of electrodes



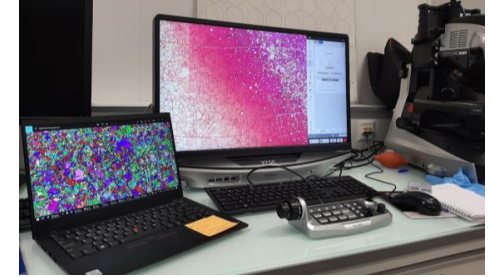
H- irradiation



LES tests

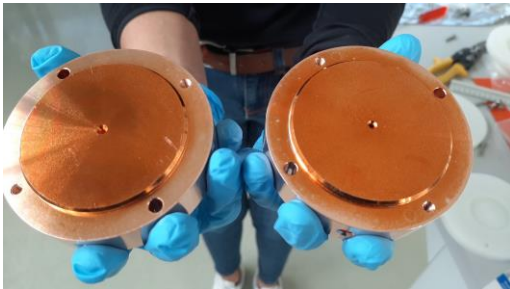


Analysis

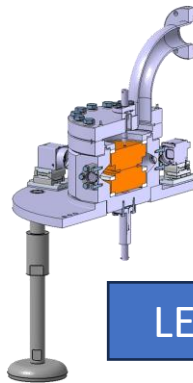


## 2<sup>nd</sup> pair of electrodes

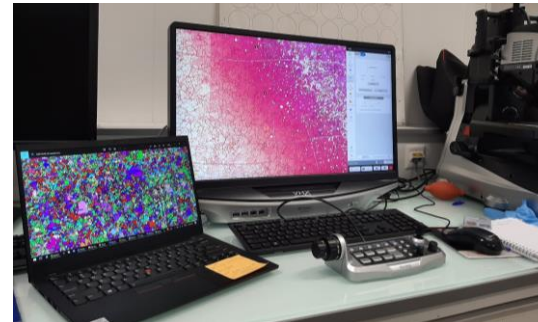
Treatment of electrodes



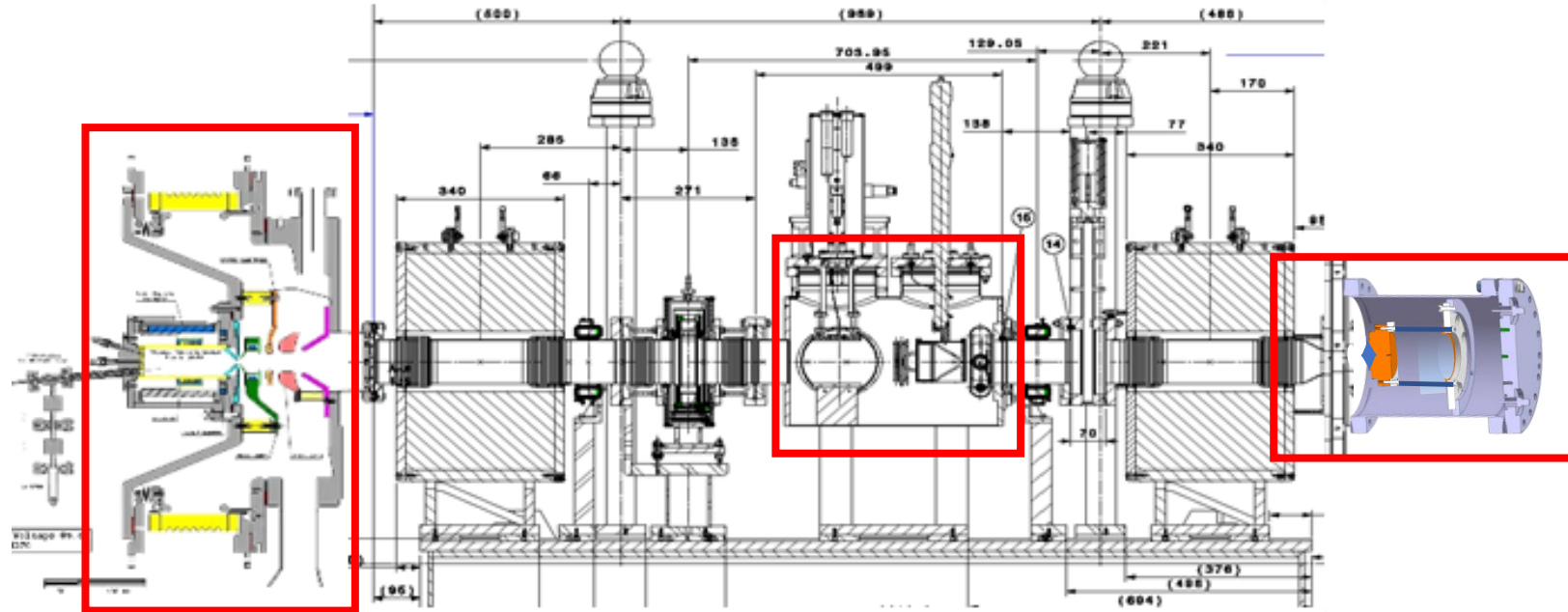
LES tests



Analysis



# Irradiation setup – LINAC4 source test stand – Irradiation affects study



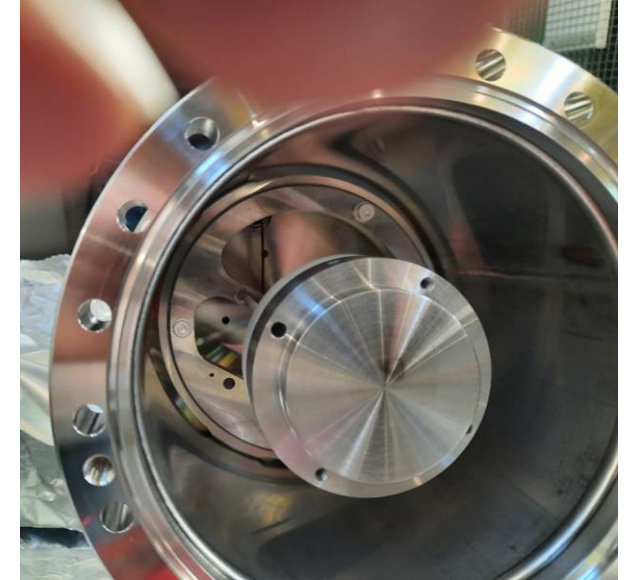
H<sup>-</sup> Source 45 keV

Low-energy beam transport

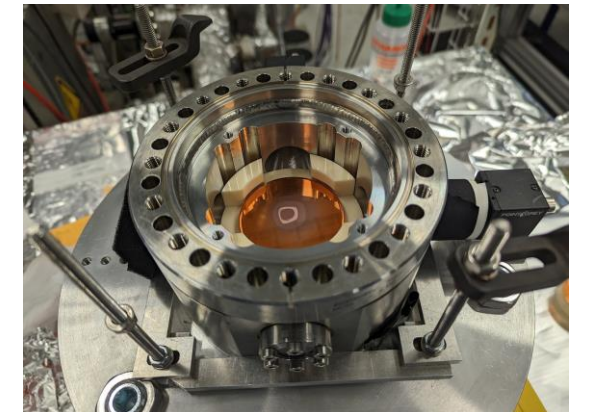
Sample holder

2 Steerers, 2 Solenoids

*Schematic from Alessandra Lombardi*

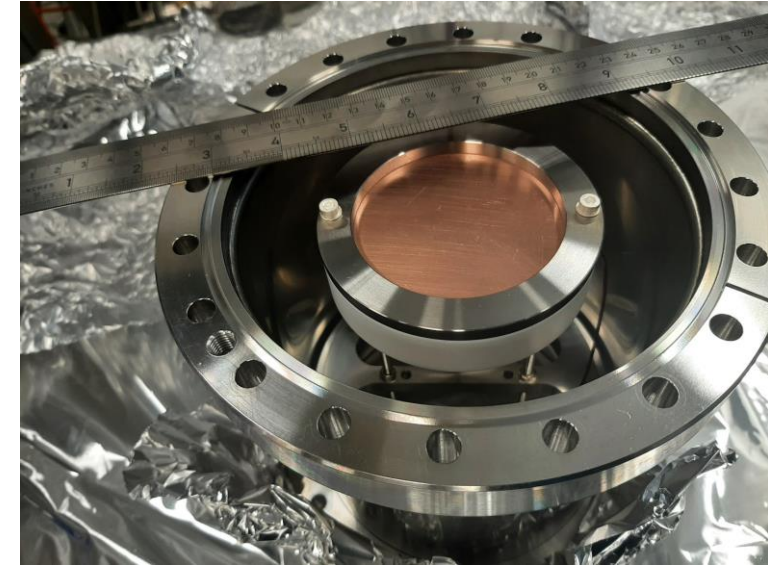
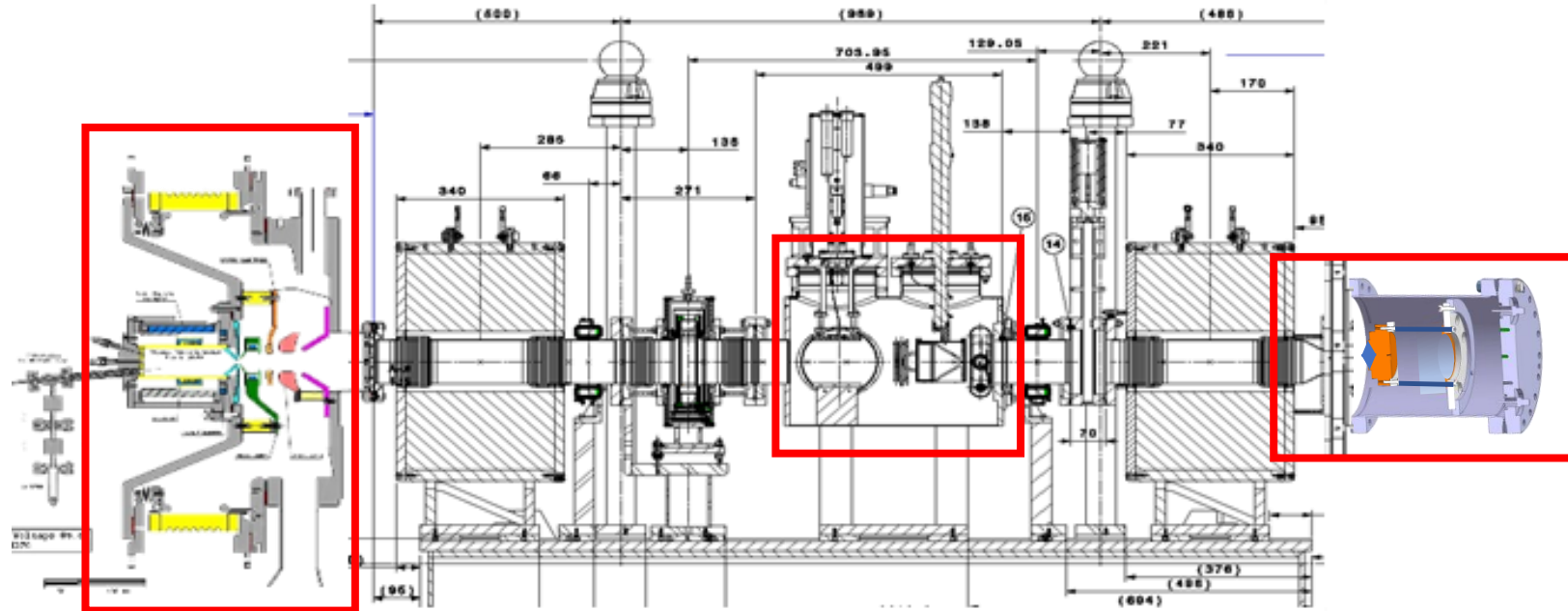


In this test stand, specific hardware was developed to use a cathode as the target for irradiation.





# Irradiation setup – LINAC4 source test stand – Irradiation affects study



H<sup>-</sup> Source 45 keV

Low-energy beam transport

2 Steerers, 2 Solenoids

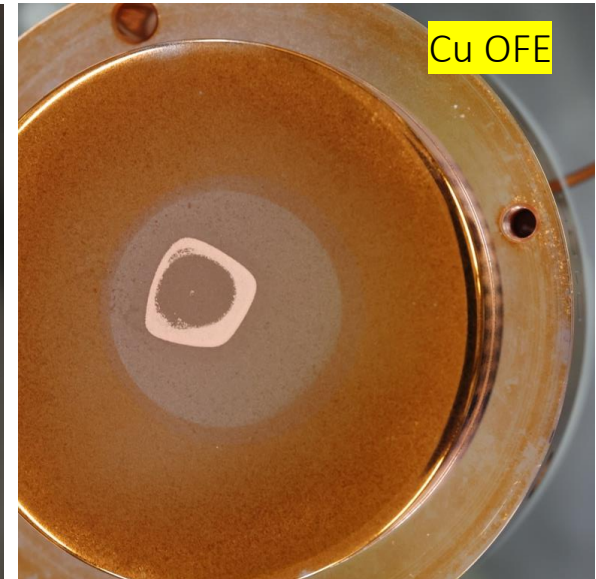
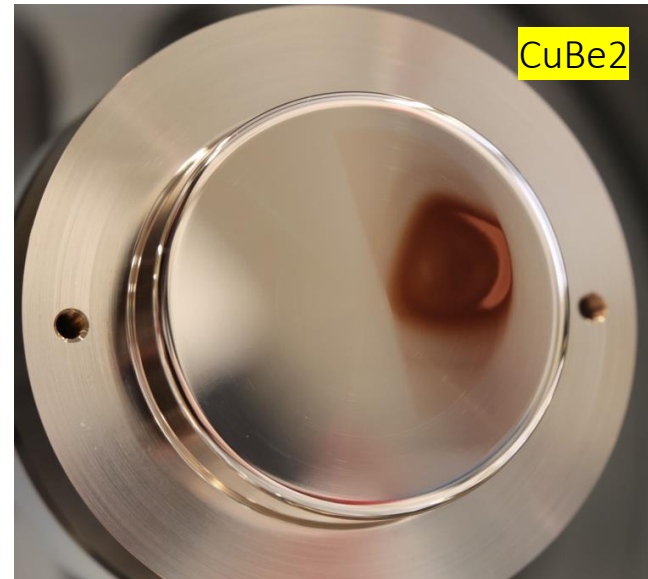
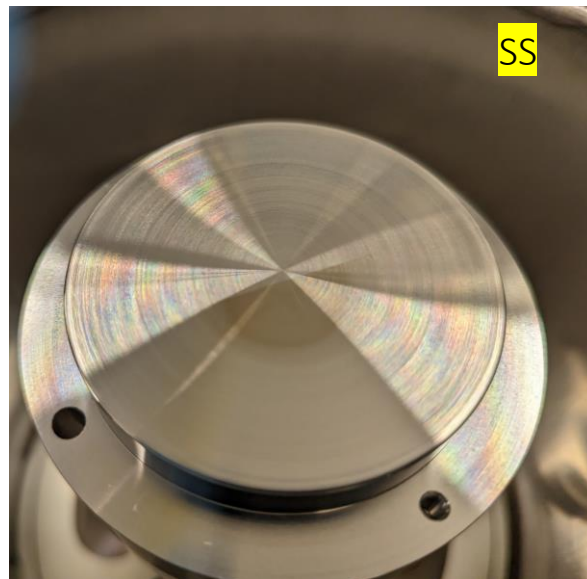
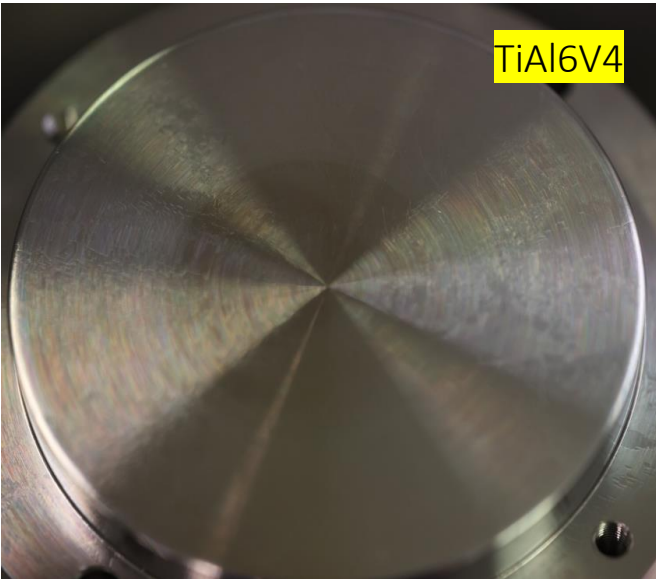
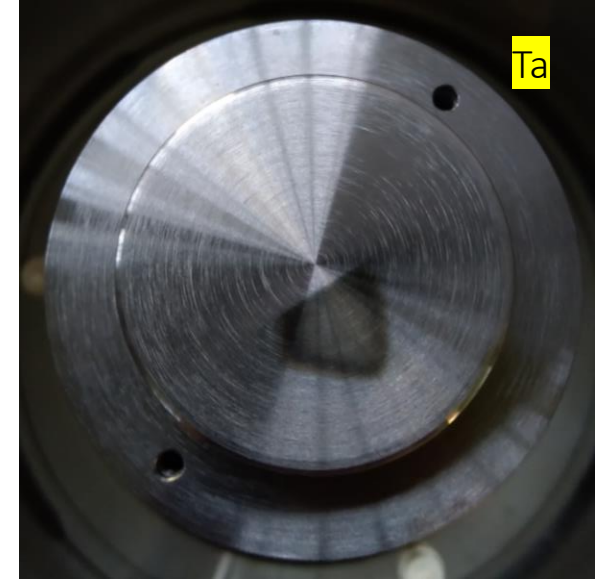
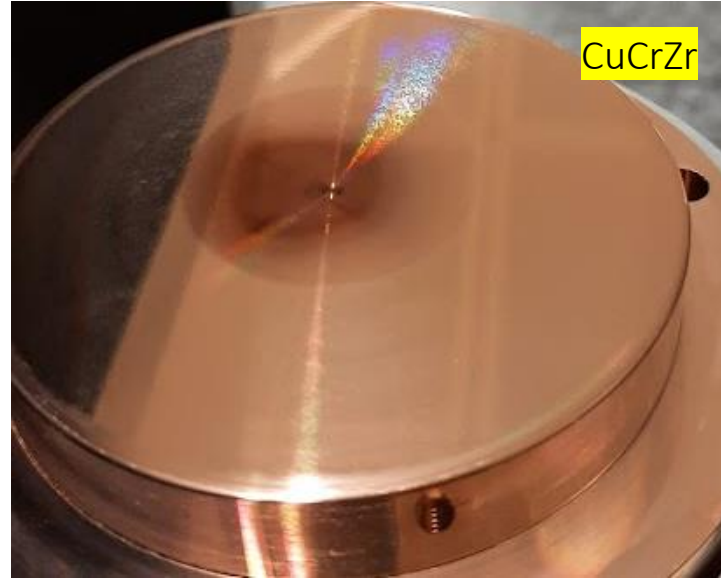
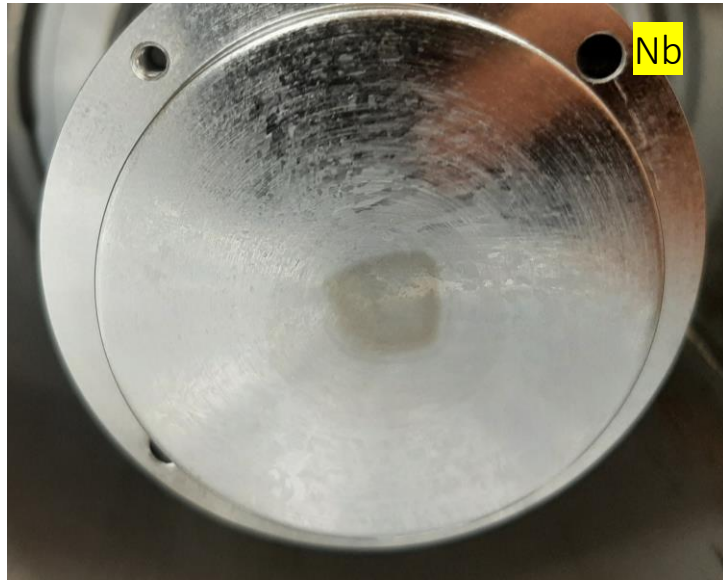
Sample holder

Low Energy H- beam	45 keV
Duration	40 hours
Pulse duration	600 μs
Repetition Rate	0.83 Hz
Peak current	20 mA
Deposition of particles on the target	1.2x 10 <sup>19</sup> H <sup>-</sup> p/cm <sup>2</sup>

Schematic from Alessandra Lombardi

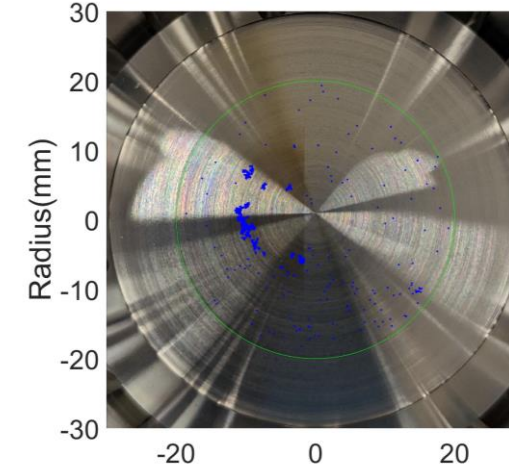
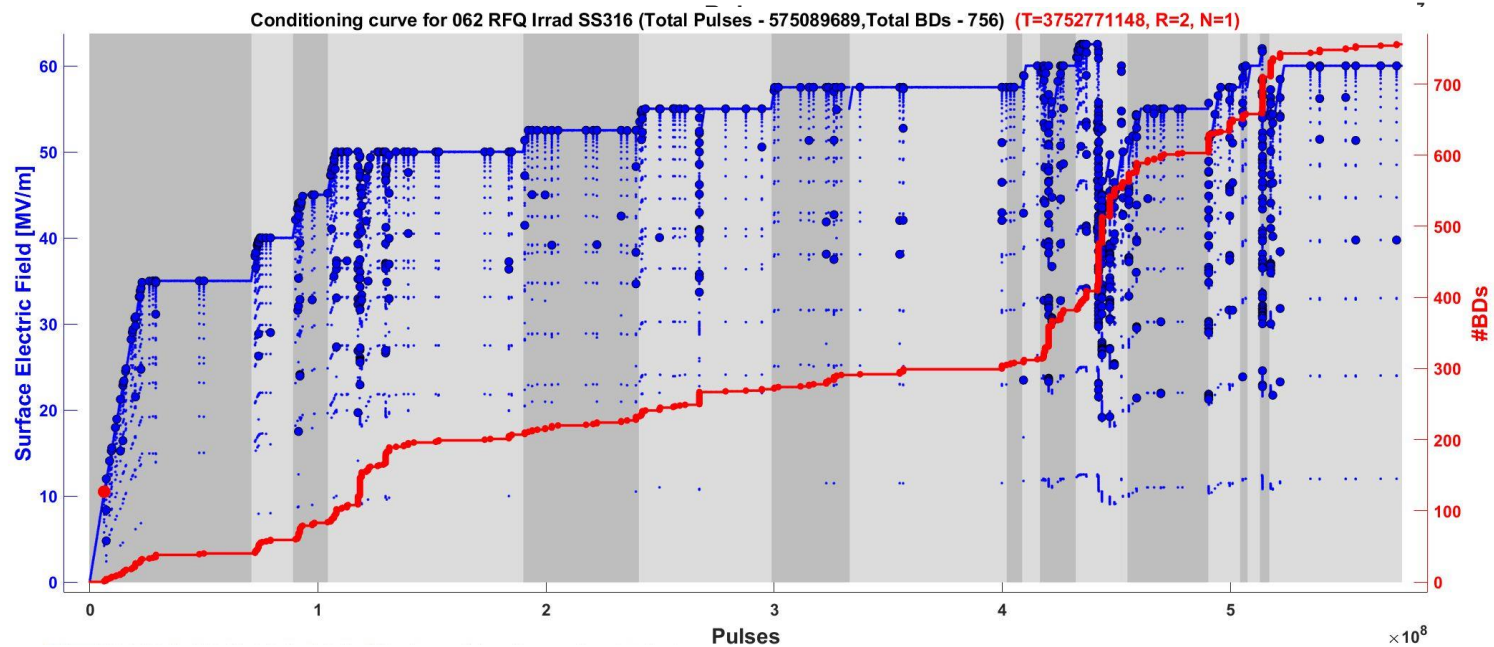
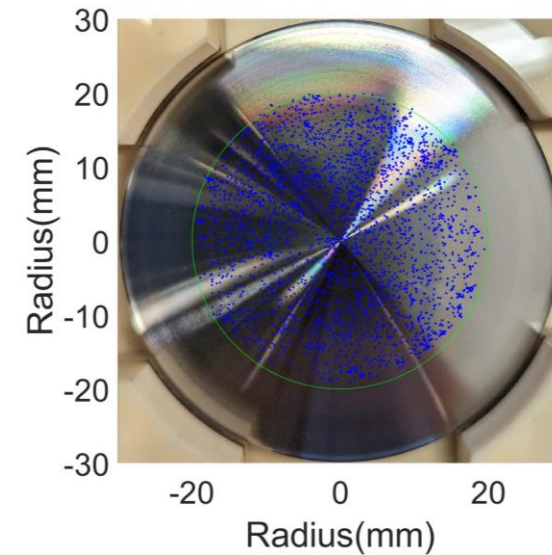
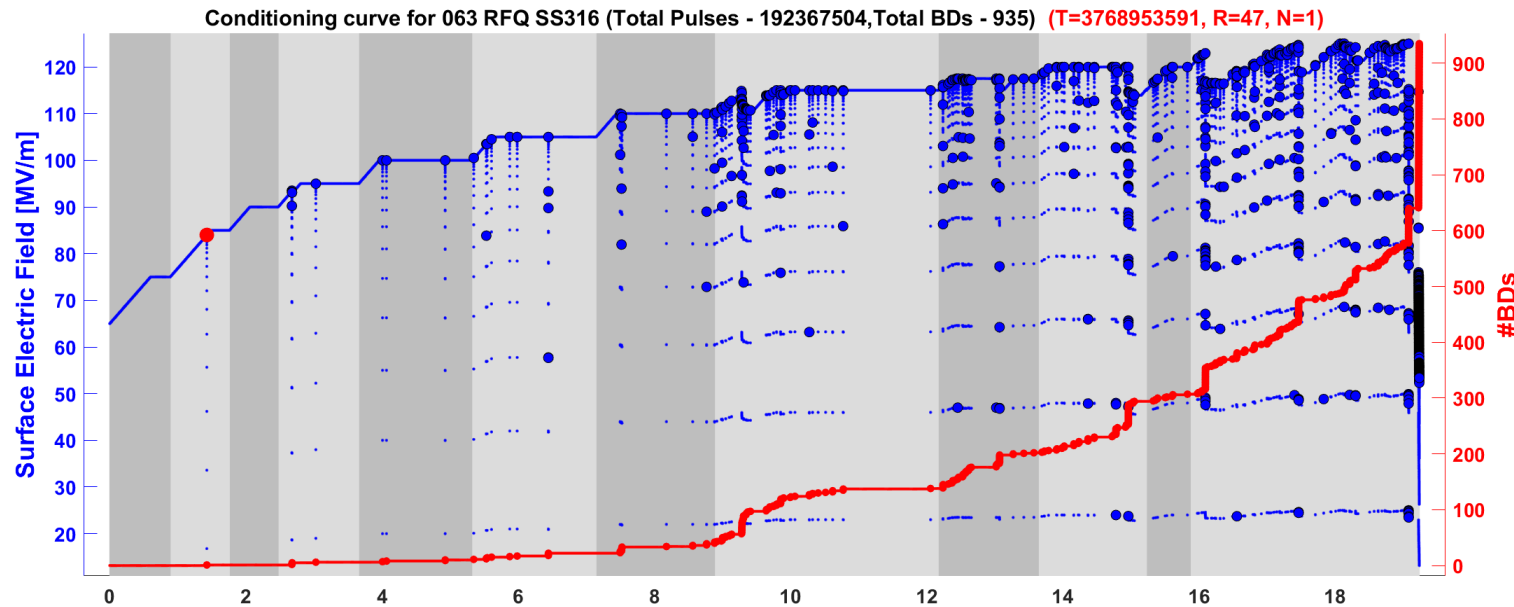


*Pictures of each electrode from each material after irradiation testing.*



**Stainless Steel electrodes non-irradiated**  
**(last 12 runs).**

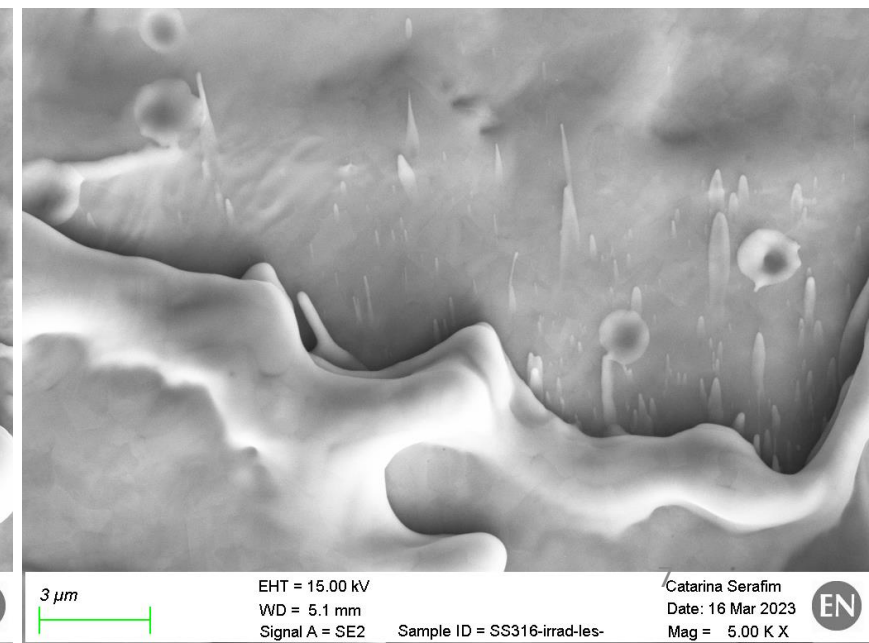
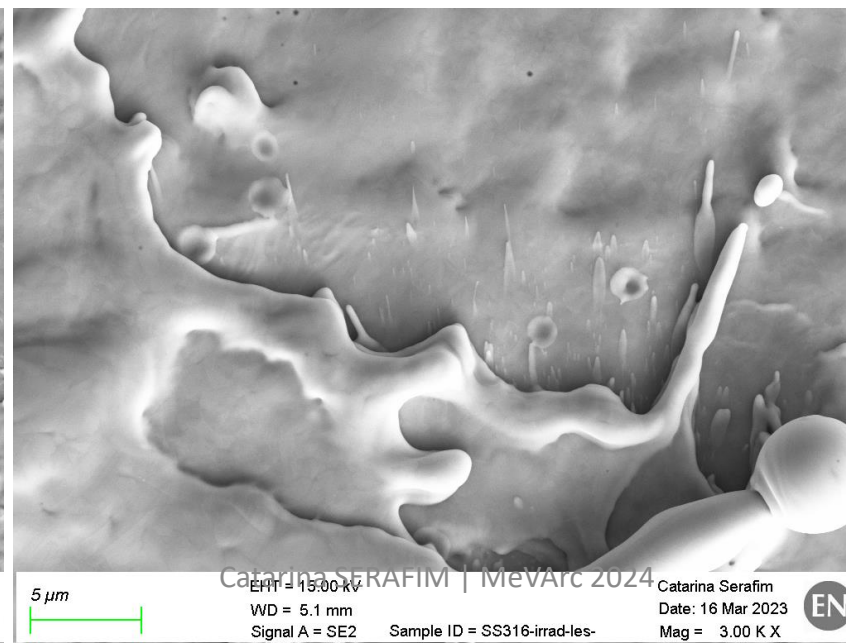
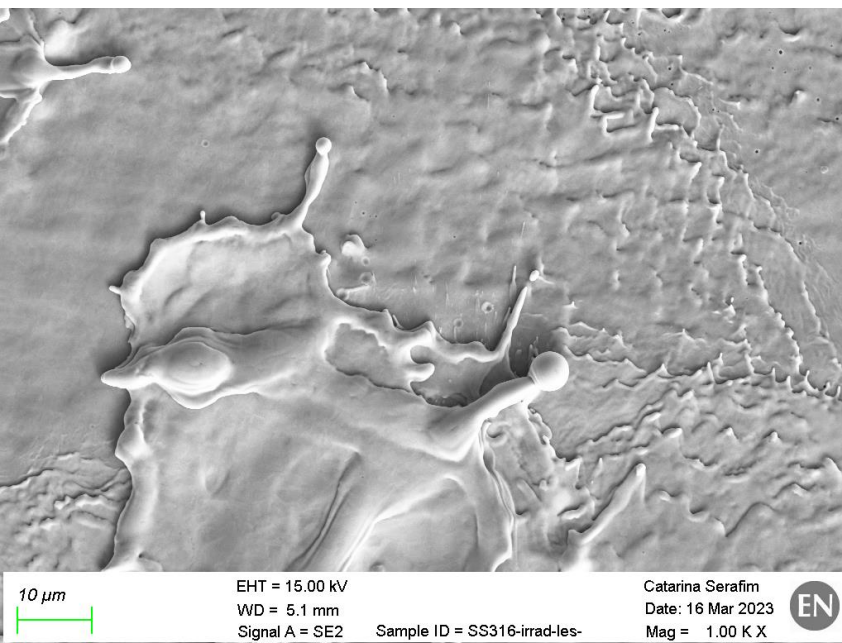
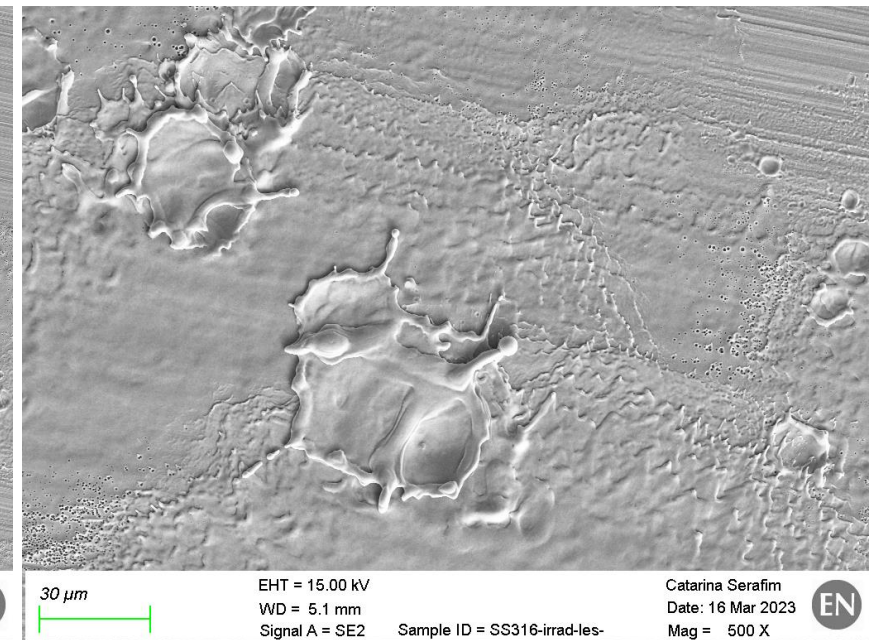
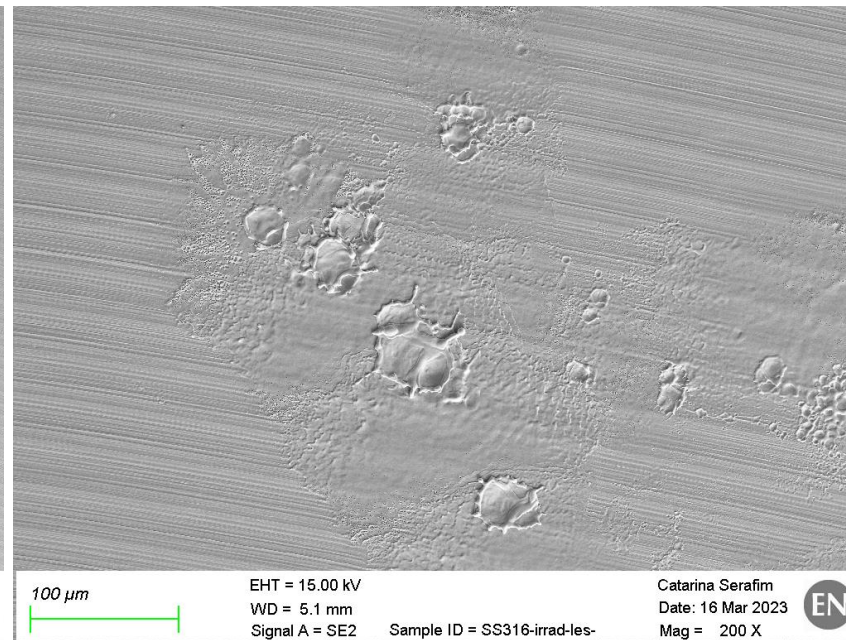
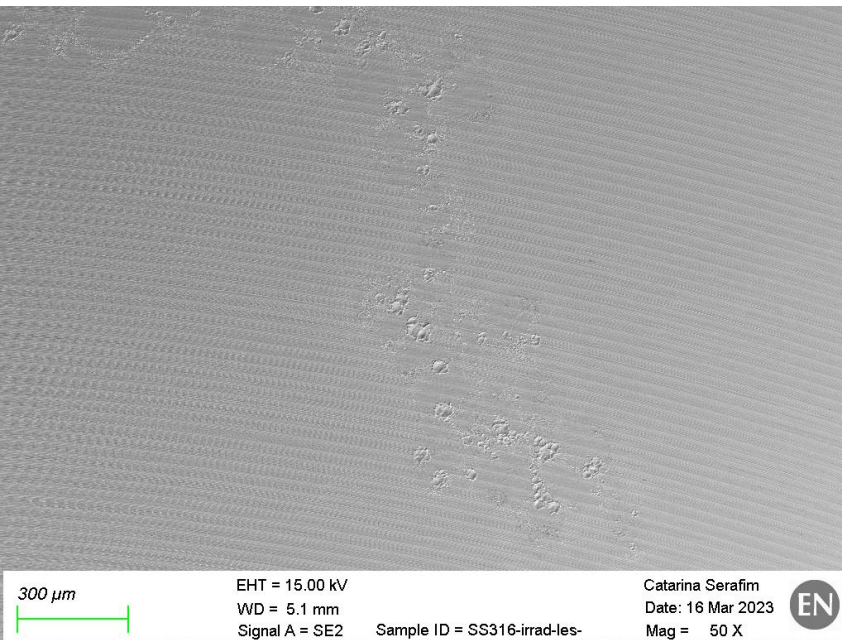
**Maximum field 120 MV/m.**



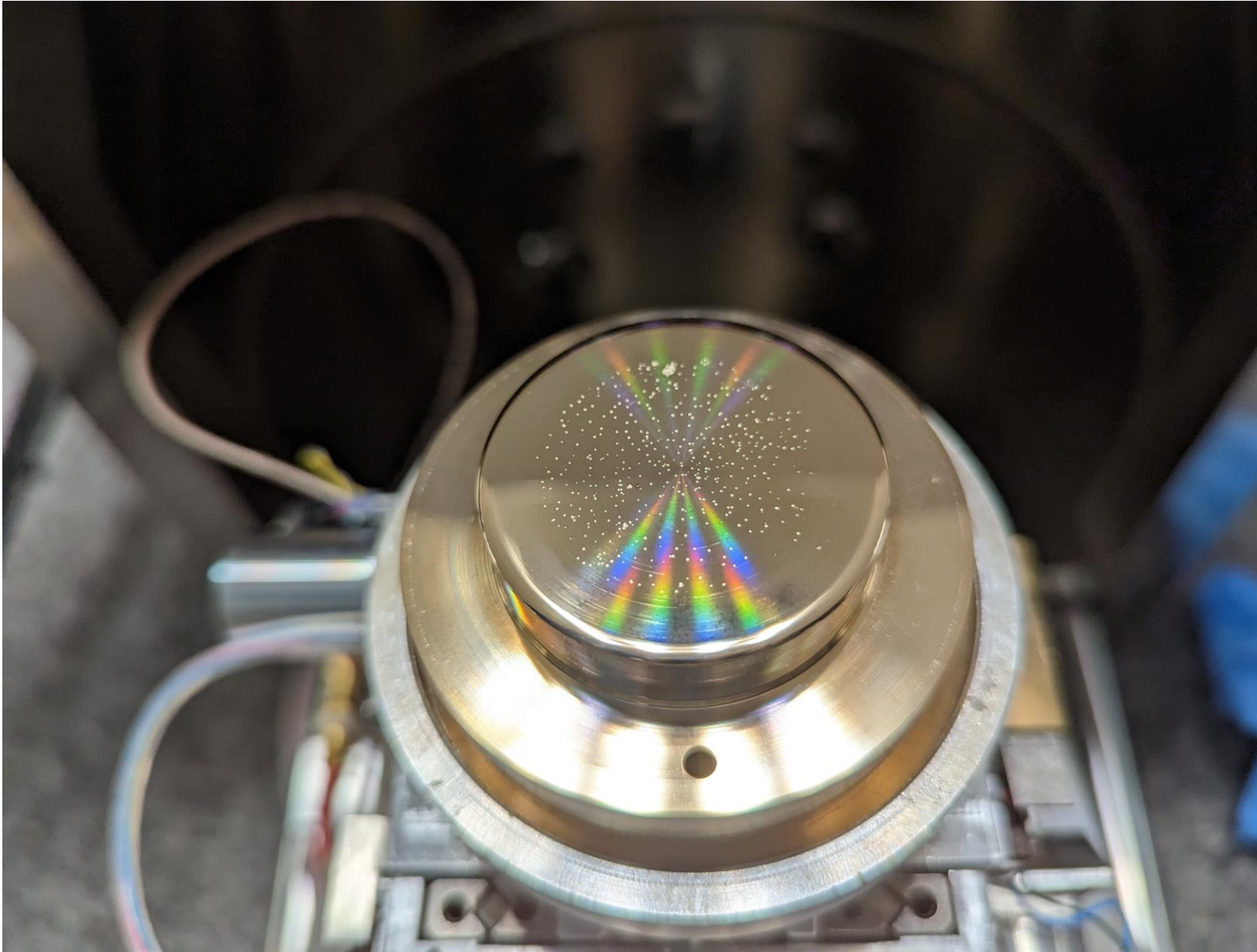
**Stainless Steel electrodes irradiated**  
**Maximum field 62 MV/m.**



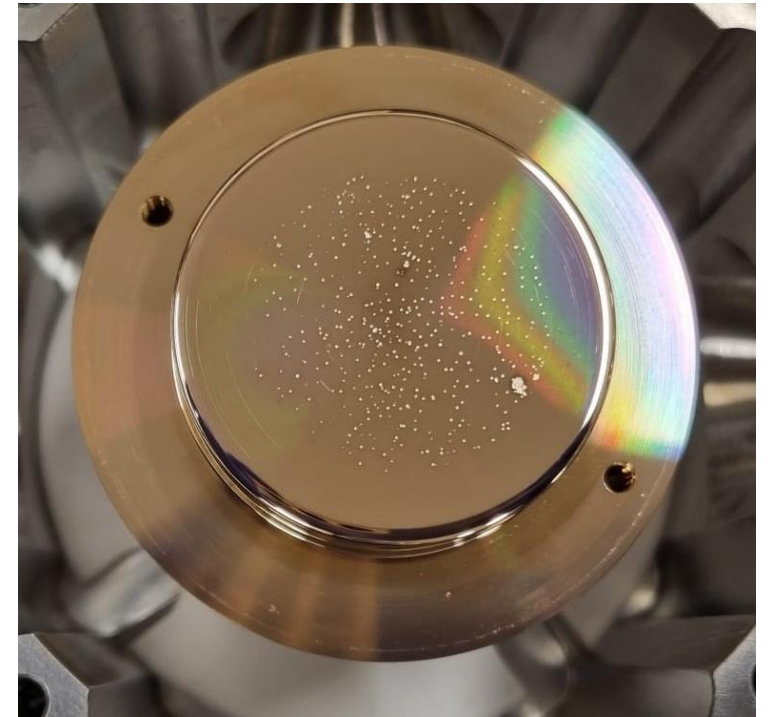
# SS316 - Irradiated Electrodes - SEM





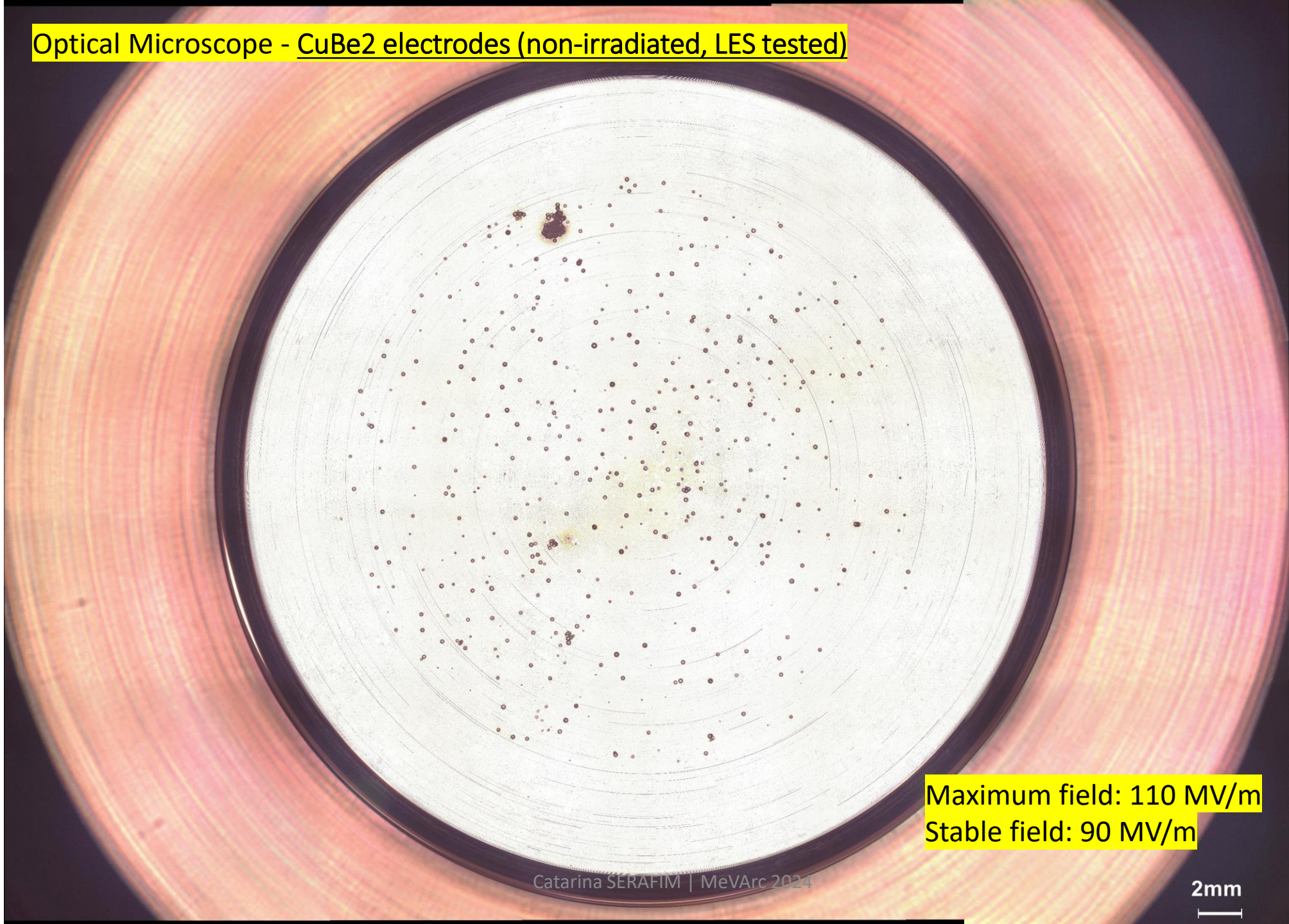


CuBe2 electrodes  
(non-irradiated, LES tested)





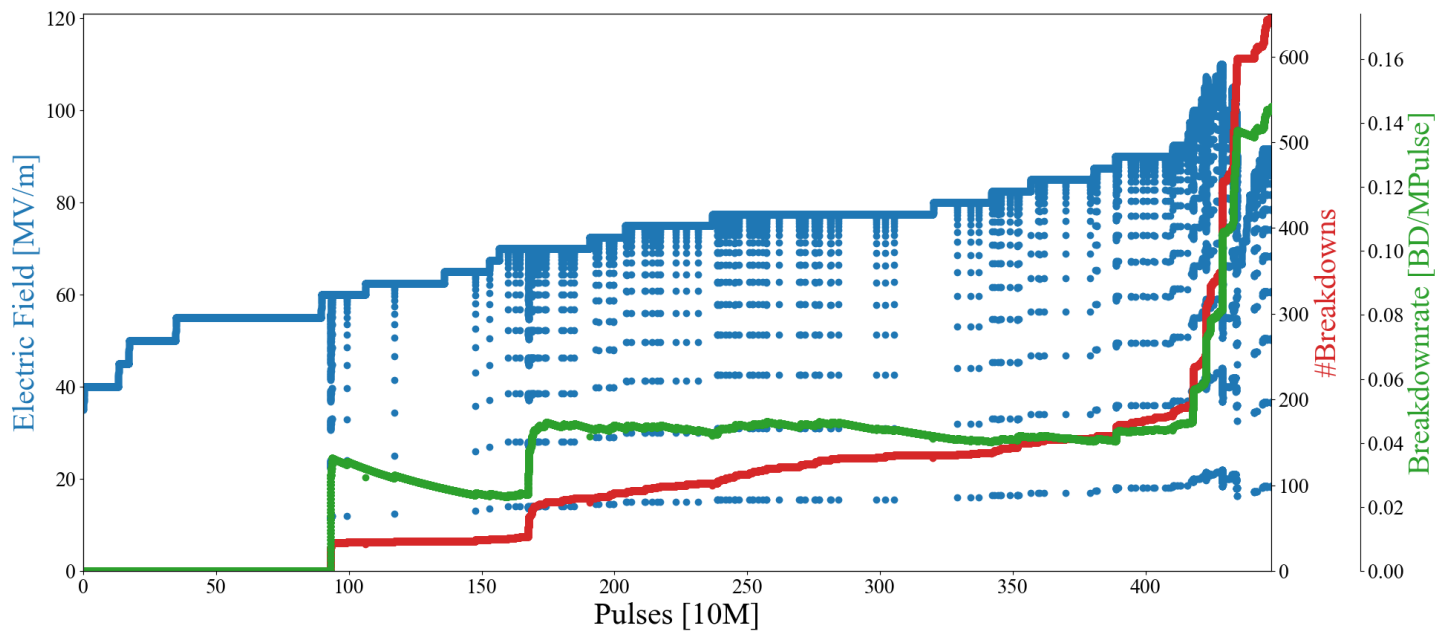
Optical Microscope - CuBe2 electrodes (non-irradiated, LES tested)



Maximum field: 110 MV/m  
Stable field: 90 MV/m

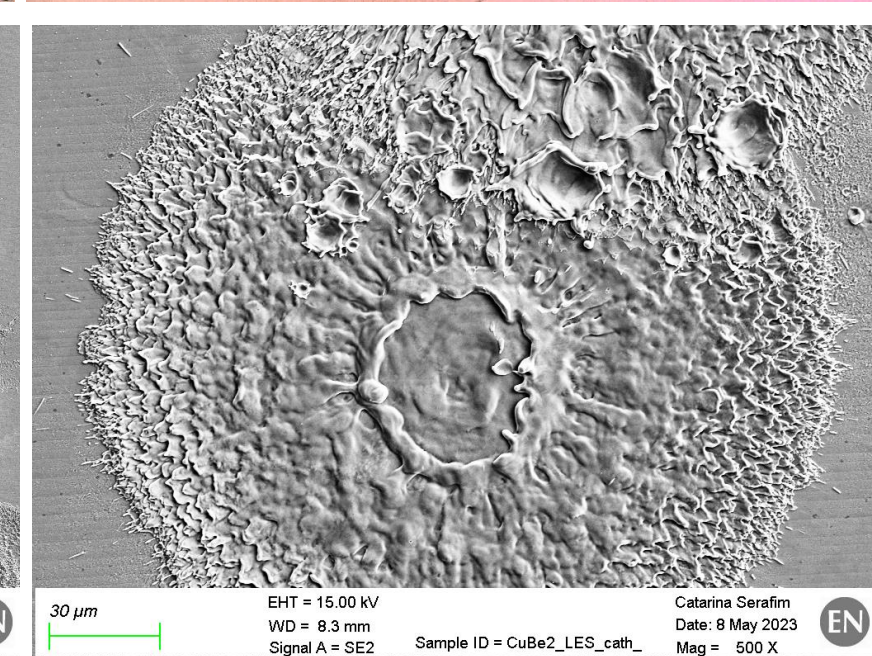
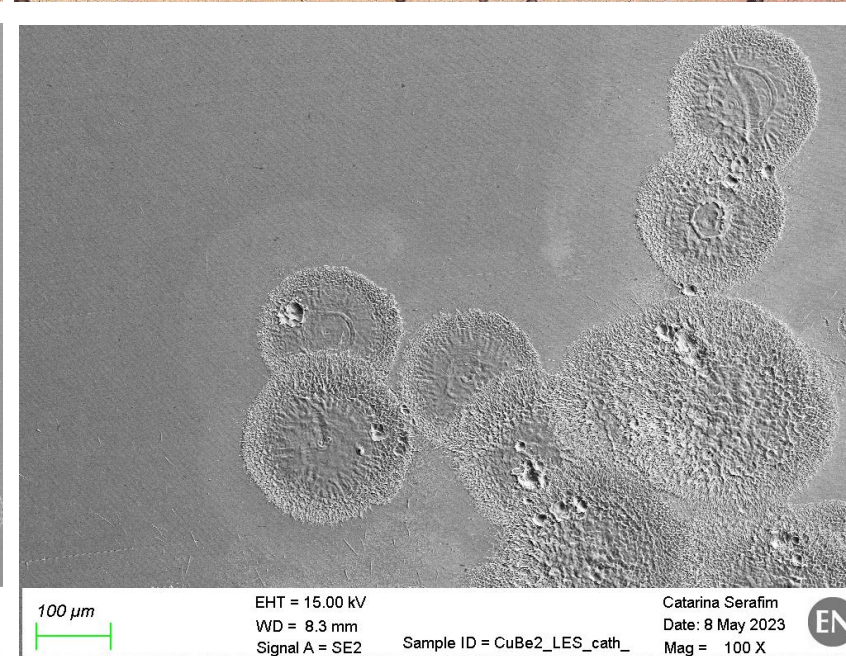
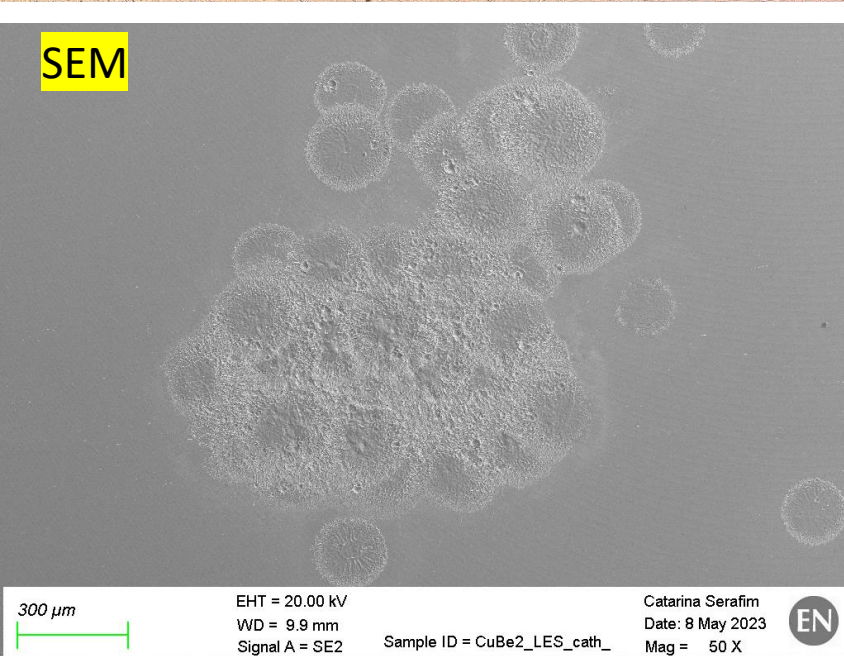
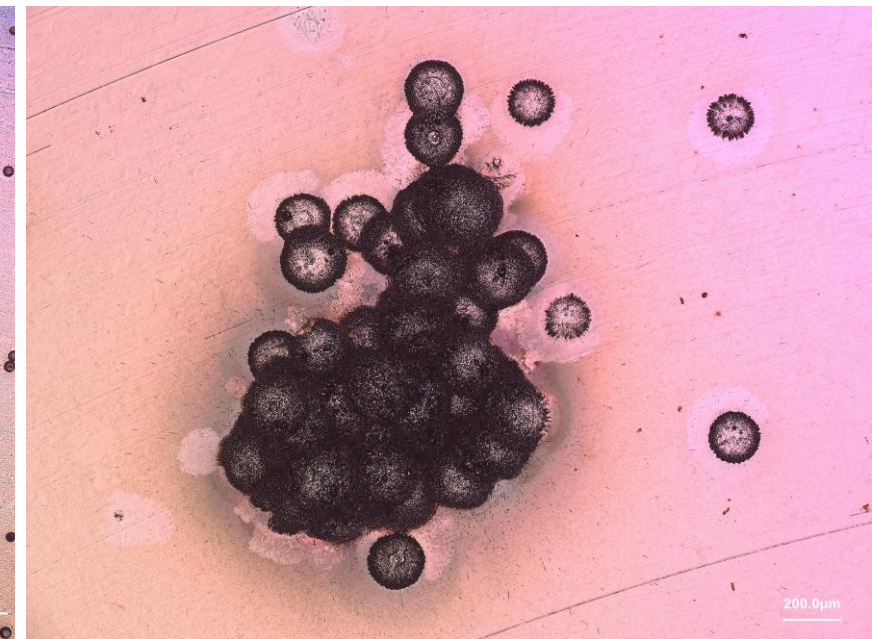
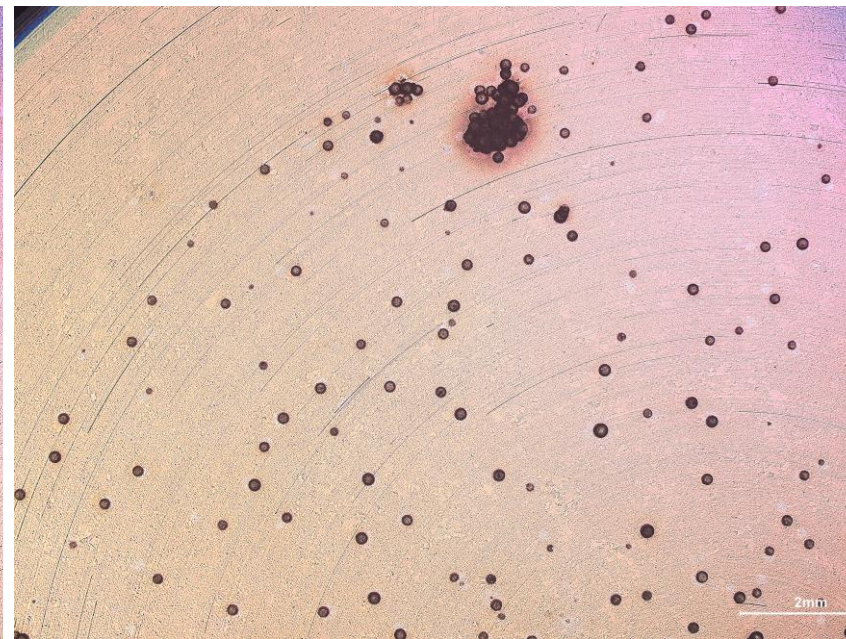
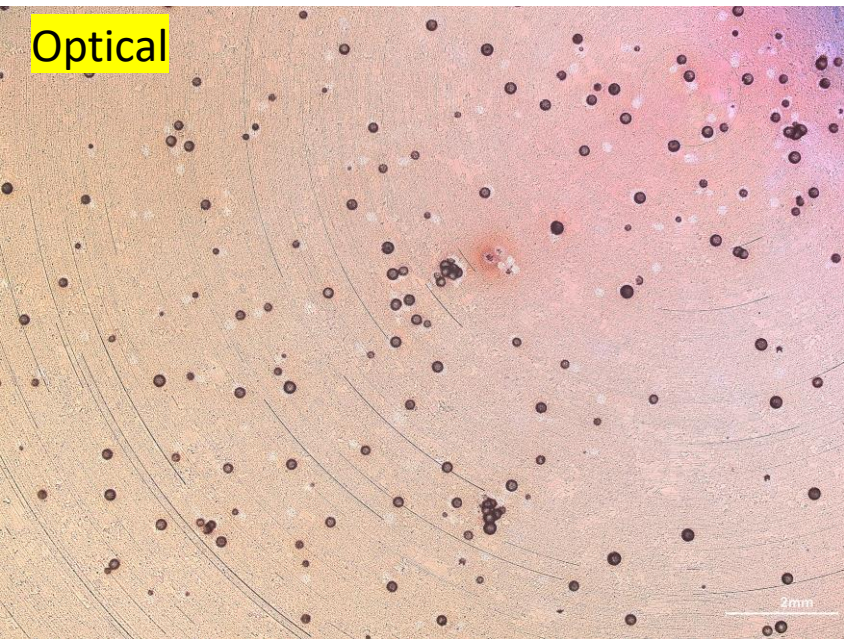


Optical Microscope - CuBe2 electrodes (non-irradiated, LES tested)



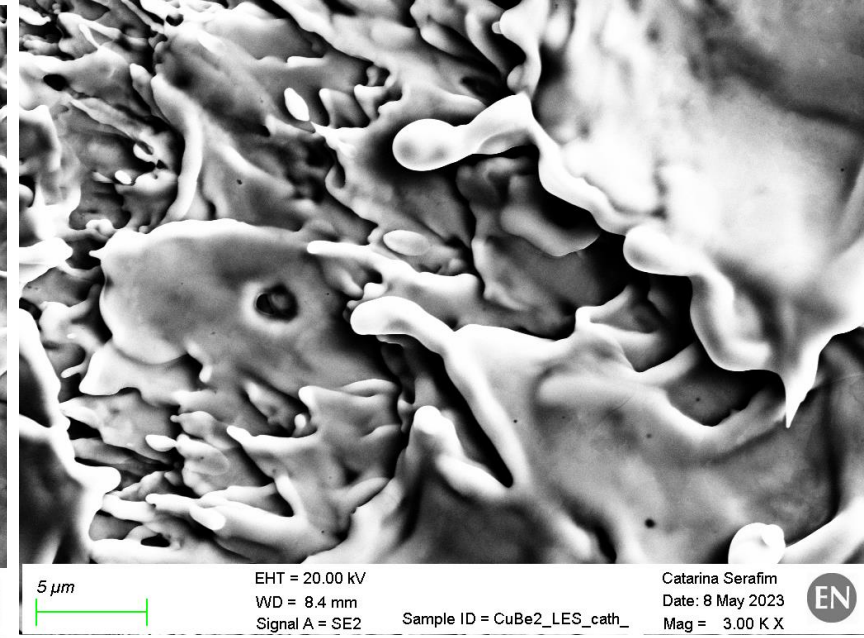
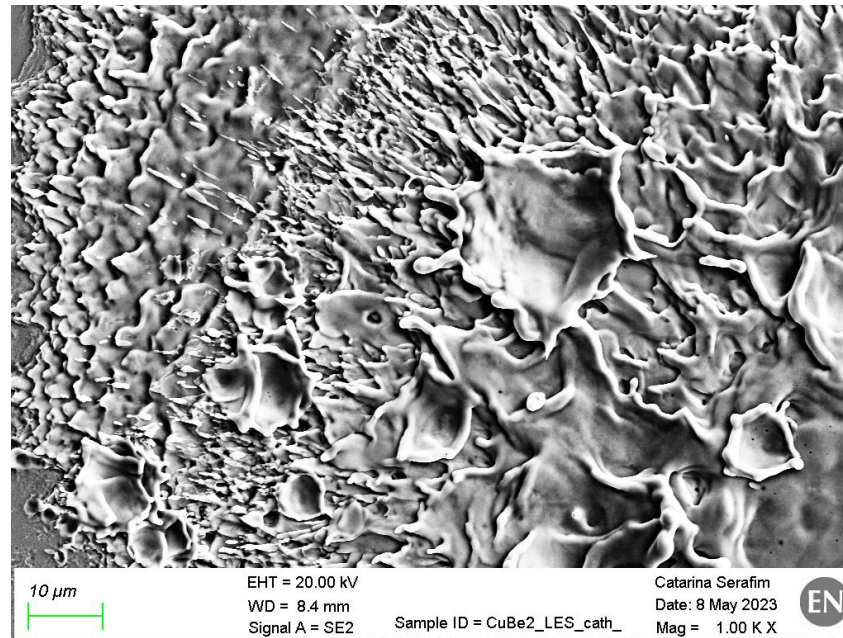
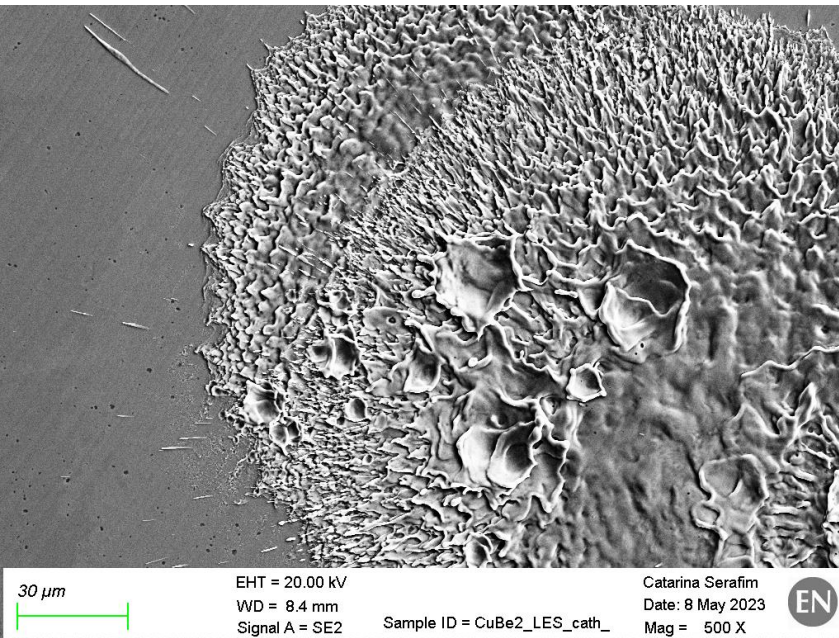
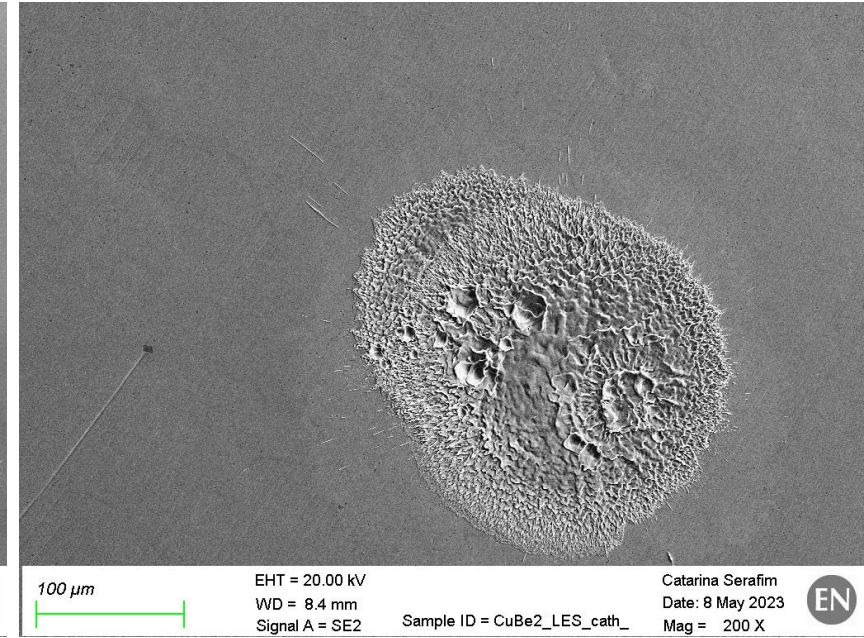
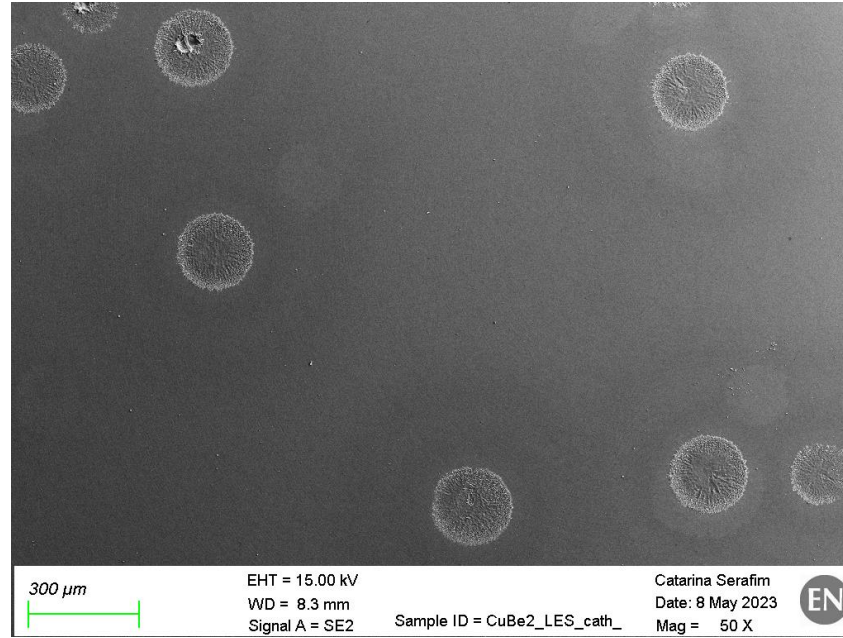
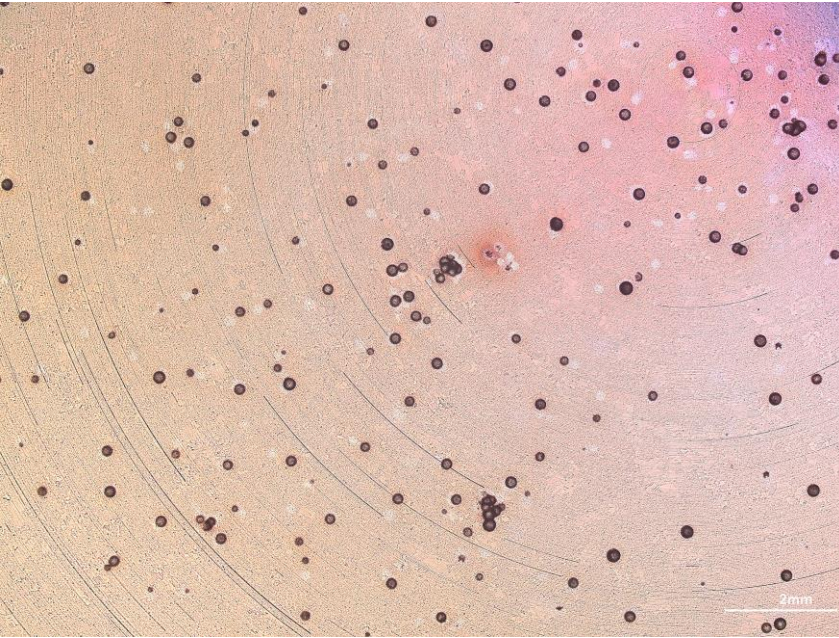
Maximum field: 110 MV/m  
Stable field: 90 MV/m





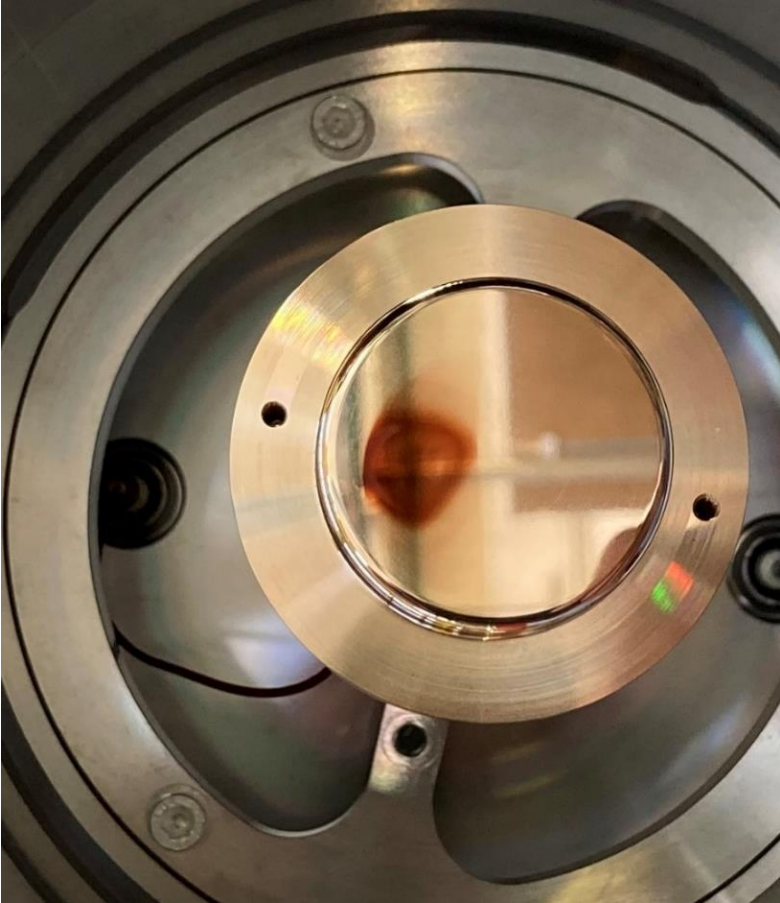


# SEM CuBe2 electrodes (non-irradiated and LES tested)

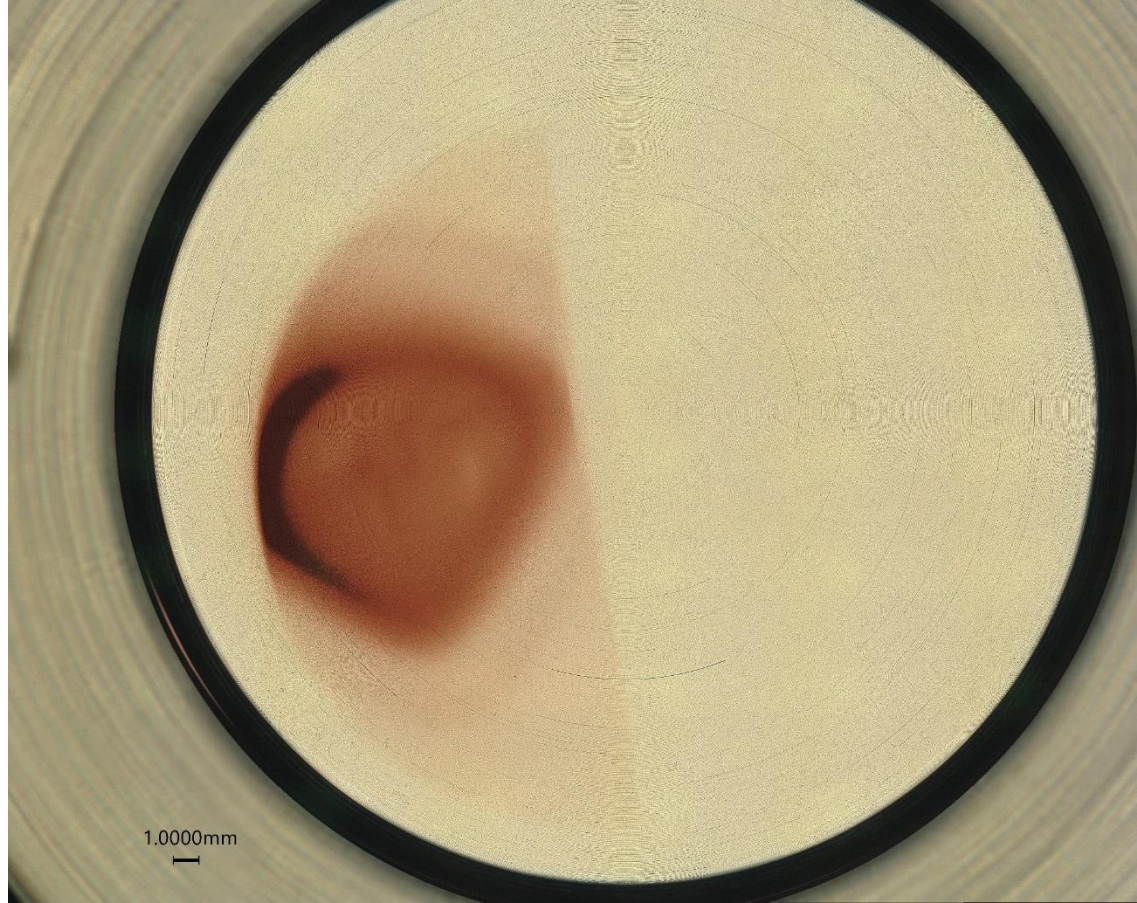




## CuBe2 cathode – irradiated



*Dismounting of CuBe cathode after irradiation*



*Optical imaging of the surface using lens 80x.*



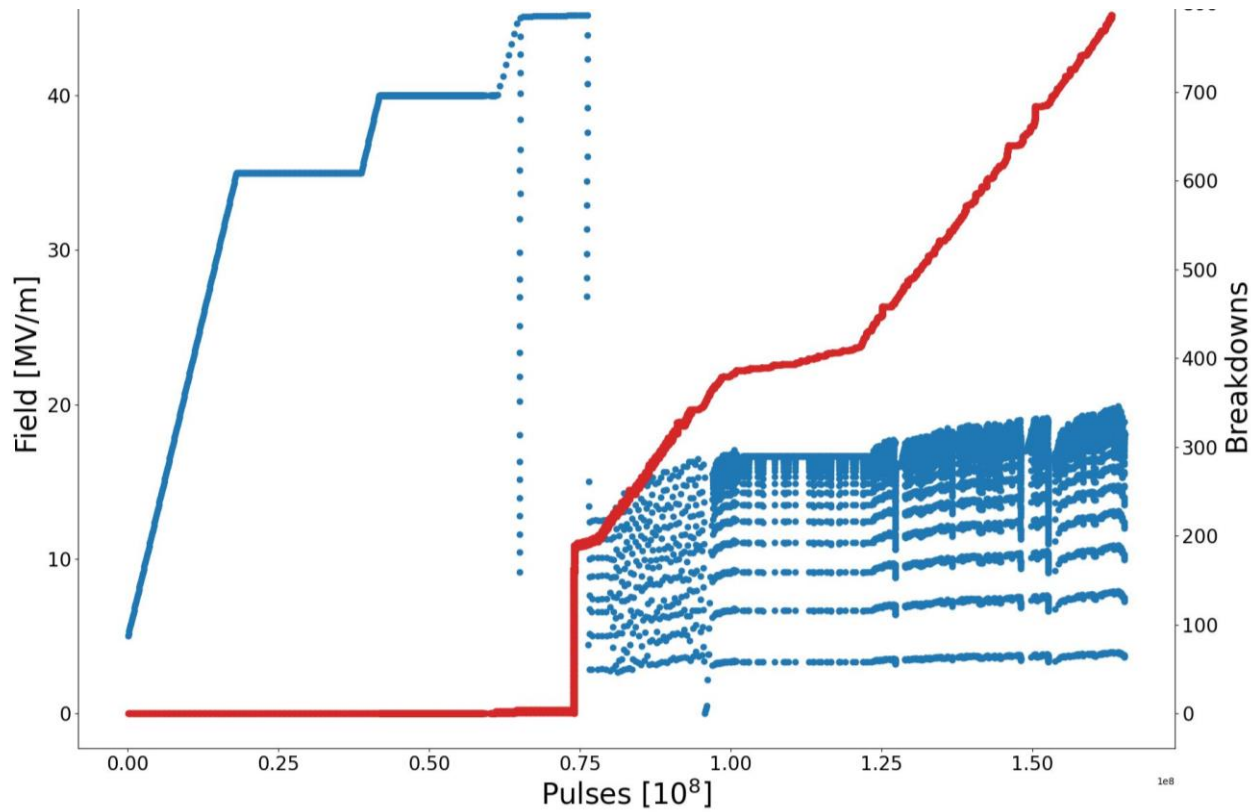
CuBe2 cathode – after irradiation



CuBe2 cathode irradiated after LES testing



This electrodes have reached a maximum field of 45 MV/m.



**CuBe2 cathode irradiated after LES testing**



**This electrodes have reached a maximum field of 45 MV/m.**

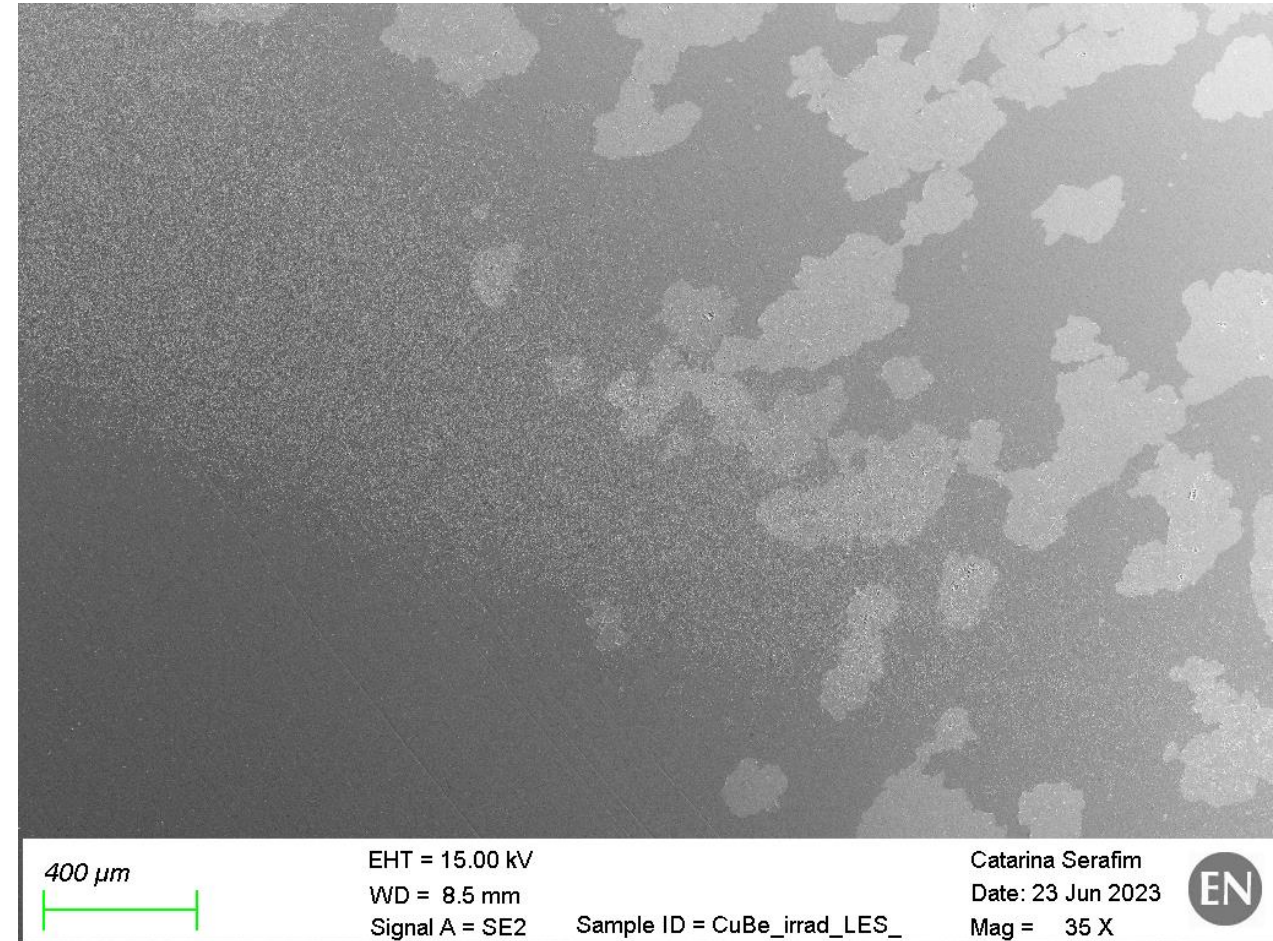
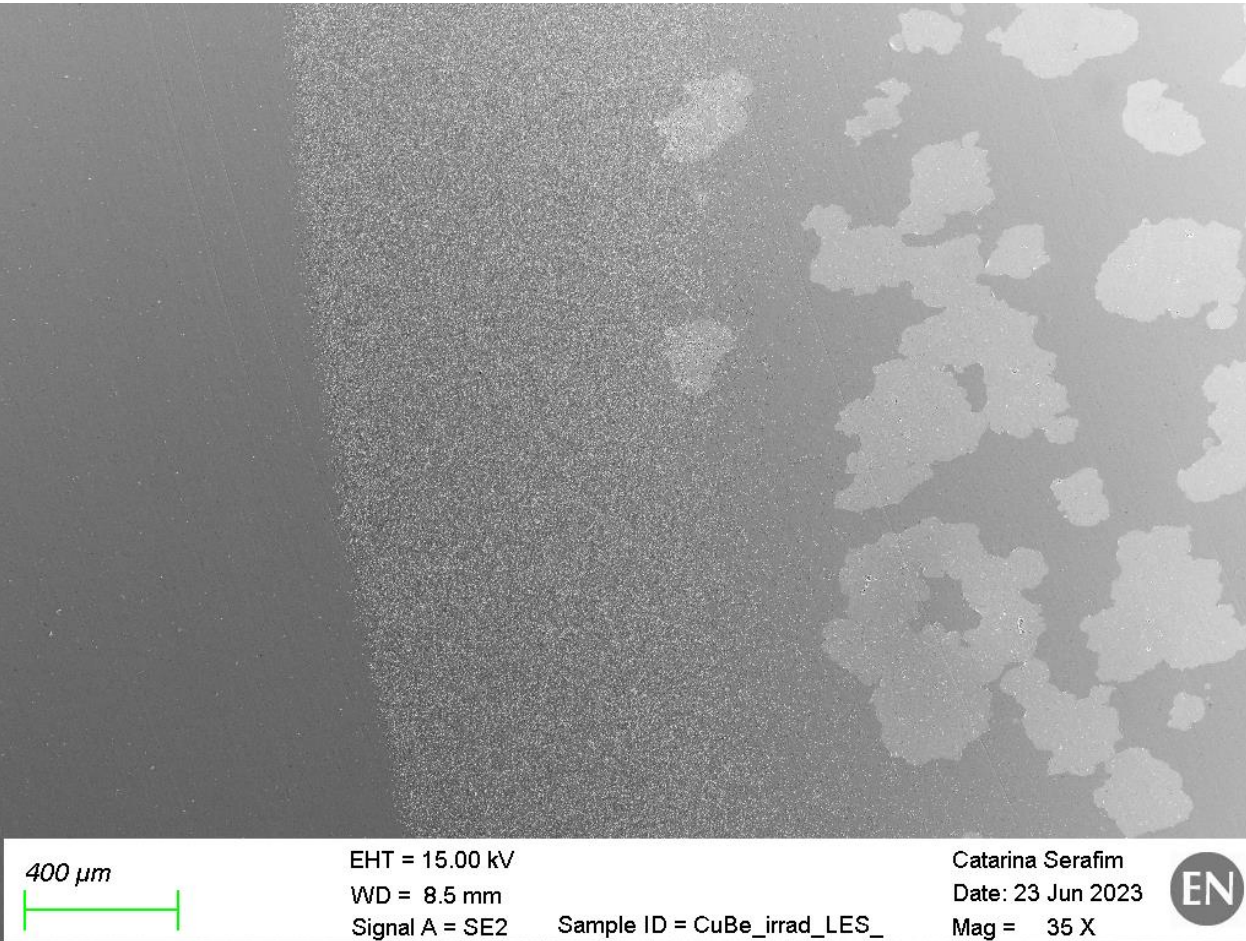
## Conclusion of LES Tests for all Materials

	Non-irradiated		Irradiated	
	Max Field (MV/m)	Stable Field (MV/m)	Max Field (MV/m)	Stable Field (MV/m)
<b>SS316LN</b>	120	120	62.5	60
<b>CuBe2</b>	110	100	40	16.7
<b>TiAl6V4</b>	110	100	95	90
<b>CuCr1Zr</b>	87.5	82.5	29	25
<b>Cu-OFE</b>	80	80	80	80
<b>Nb (BCP)</b>	94	80	42	21.7
<b>Ta</b>	60	60	38.1	24

- ✓ Cu-OFE, TiAl6V4, CuBe2 and SS316 seem to be the best materials in reaching a stable field.
- ✓ We see very big differences in the fields reached between irradiated pairs and non irradiated pairs.
- ✓ Carbon deposition from irradiation seems to be directly correlated with breakdowns appearance.
- ✓ The blistering effect from irradiation doesn't seem to provoke a decrease of performance on the electrodes.



The following images are from the irradiated cathode, submitted to high pulsing test. Similar to previous irradiations, we have observed a carbon content inside the irradiated zone, around 10%. In the irradiated zone, exfoliation of this carbon layer was observed (white spots).





Breakdowns are very small due to the low field achieved.

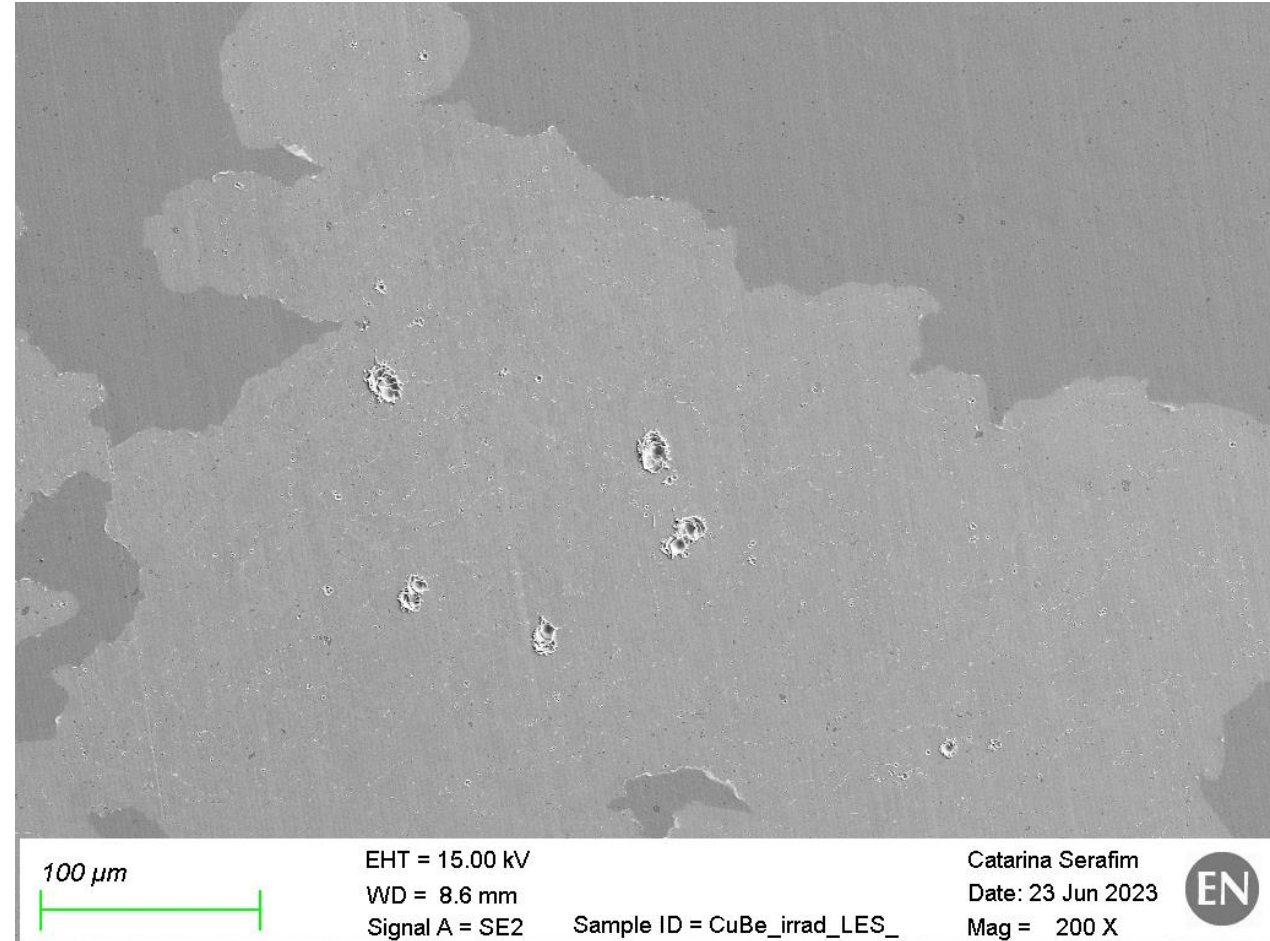
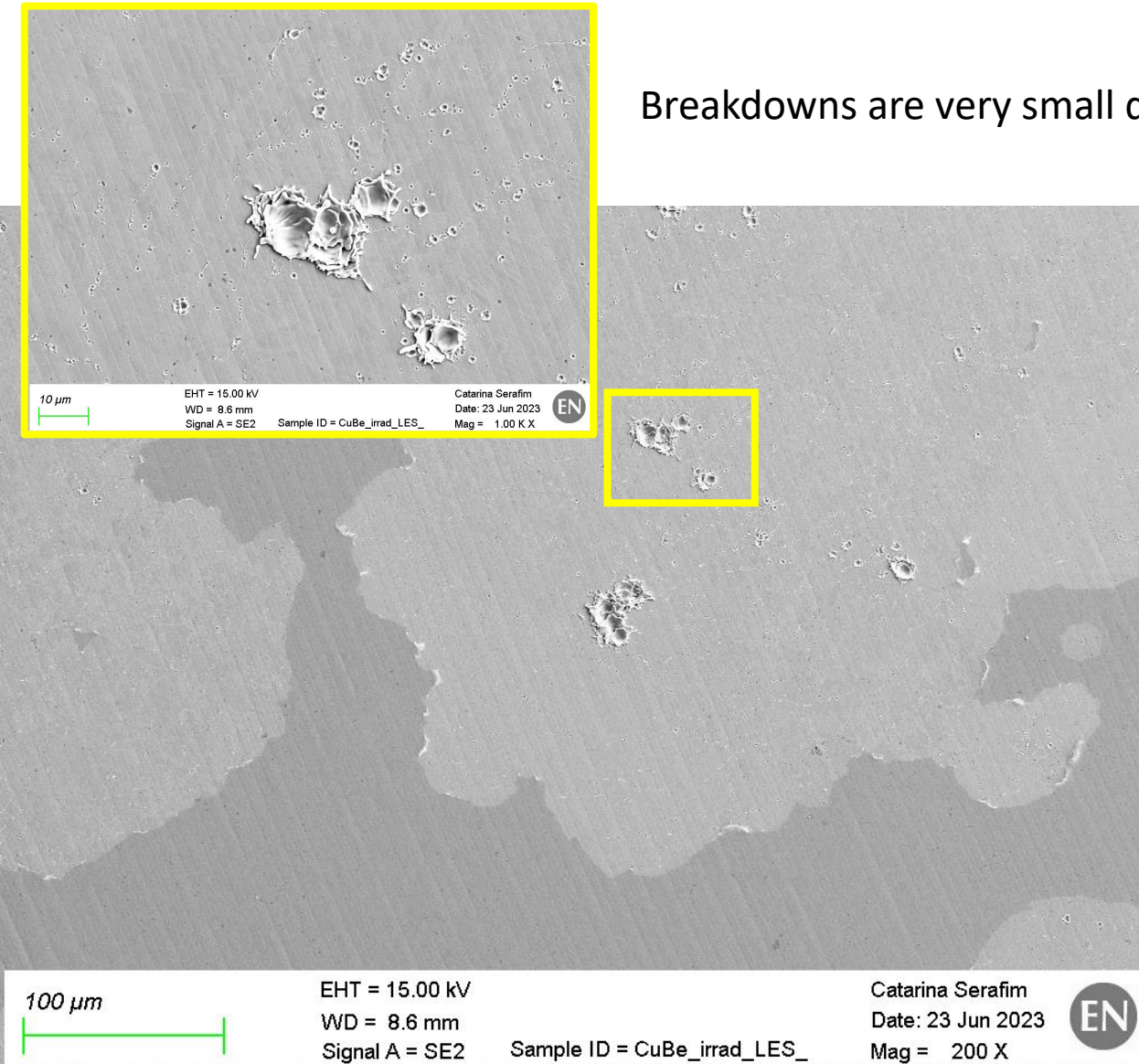
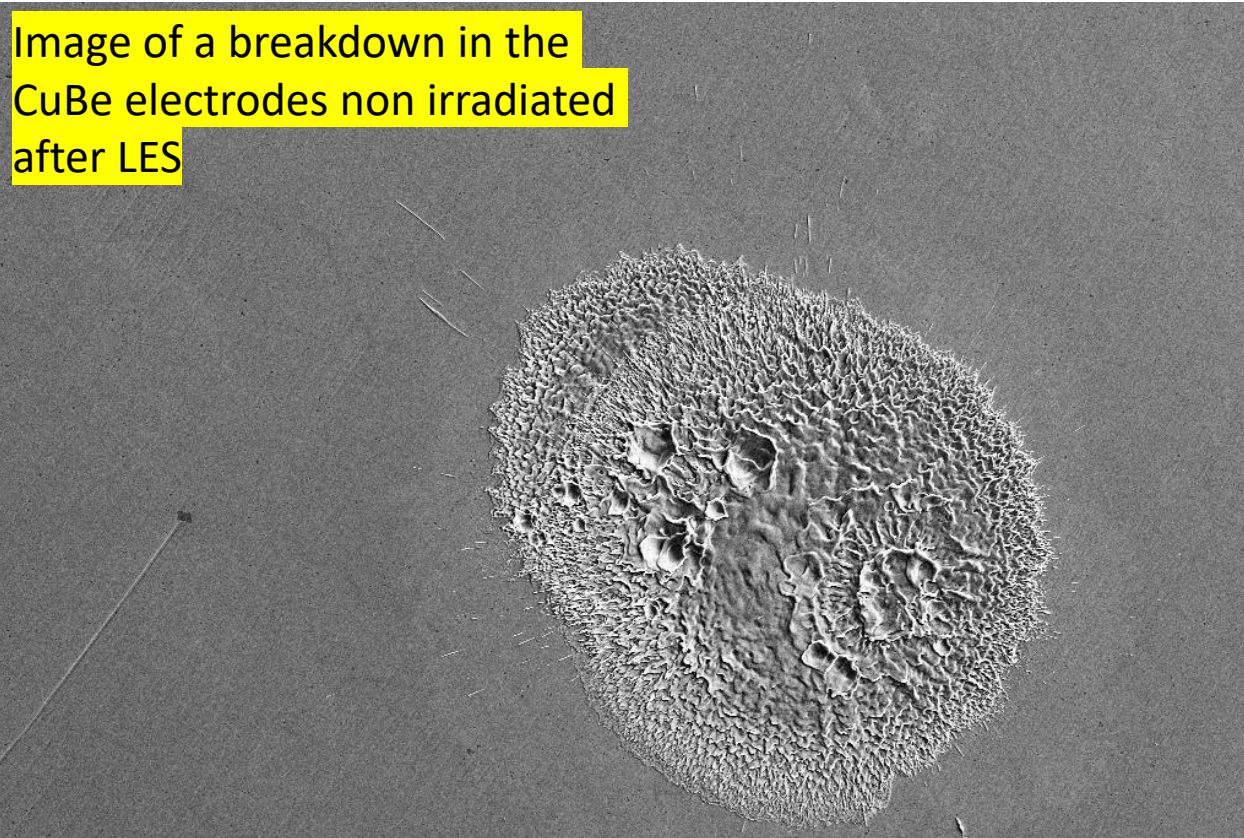


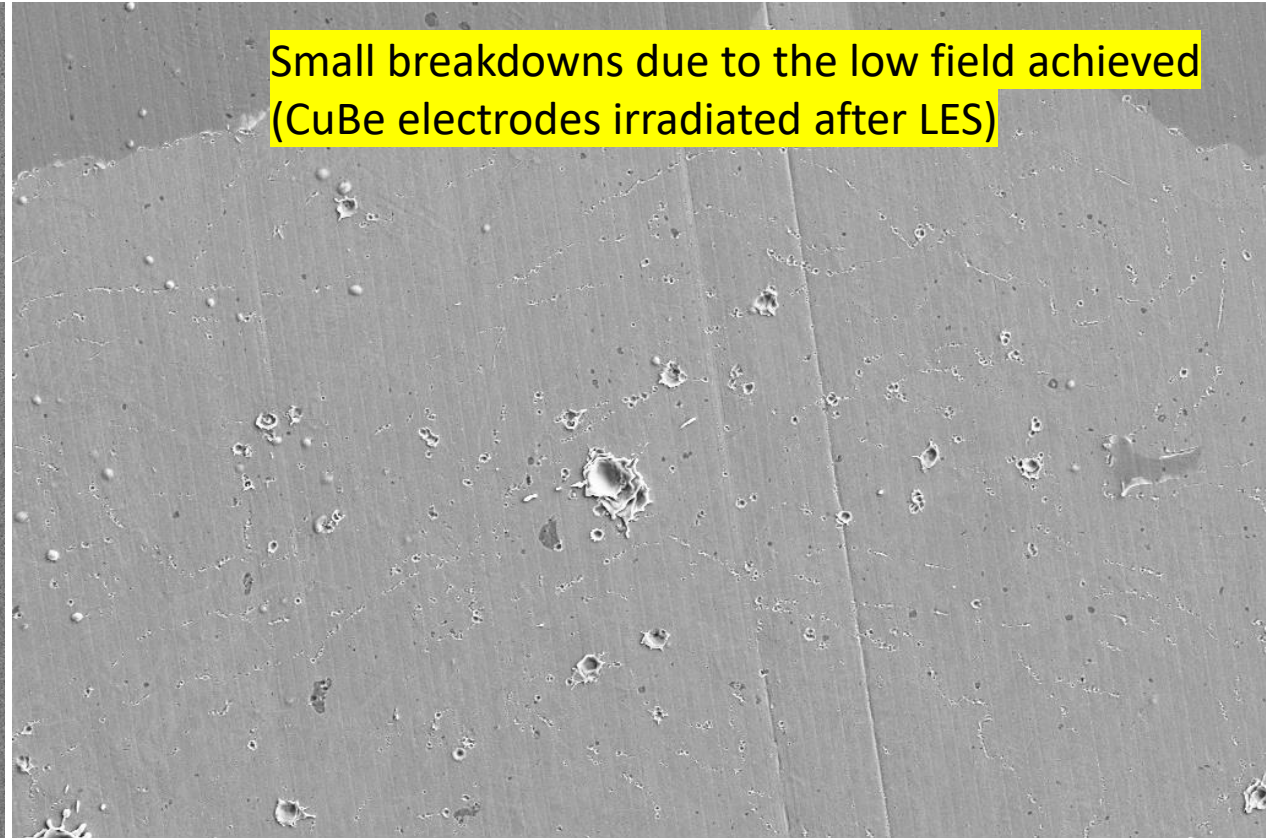


Image of a breakdown in the CuBe electrodes non irradiated after LES



100  $\mu\text{m}$  EHT = 20.00 kV  
WD = 8.4 mm Signal A = SE2  
Sample ID = CuBe2\_LES\_cath\_ Catarina Serafim  
Date: 8 May 2023 Mag = 200 X EN

Small breakdowns due to the low field achieved (CuBe electrodes irradiated after LES)

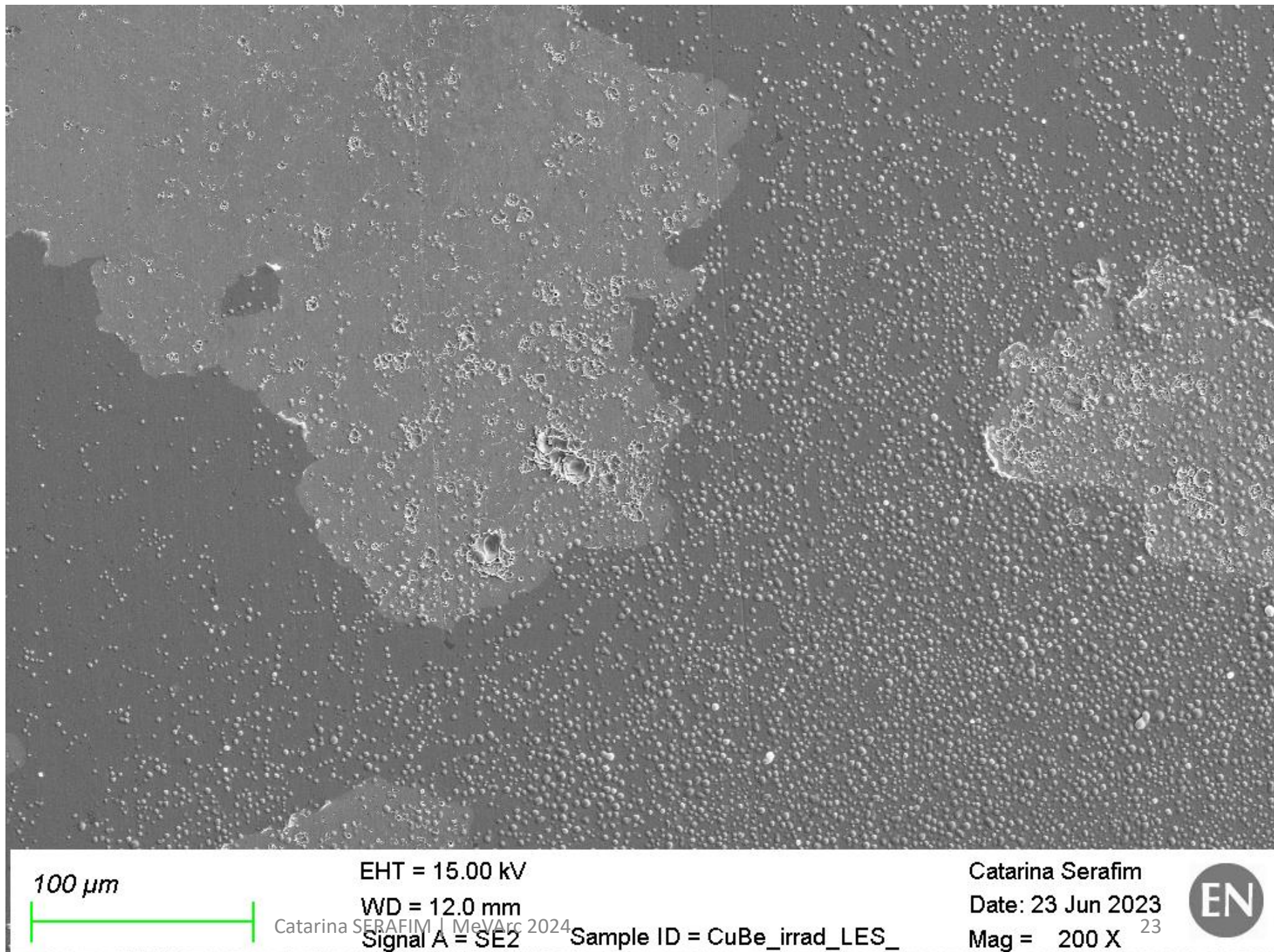


30  $\mu\text{m}$  EHT = 15.00 kV  
WD = 8.6 mm Signal A = SE2  
Sample ID = CuBe\_irrad\_LES\_ Catarina Serafim  
Date: 23 Jun 2023 Mag = 500 X EN



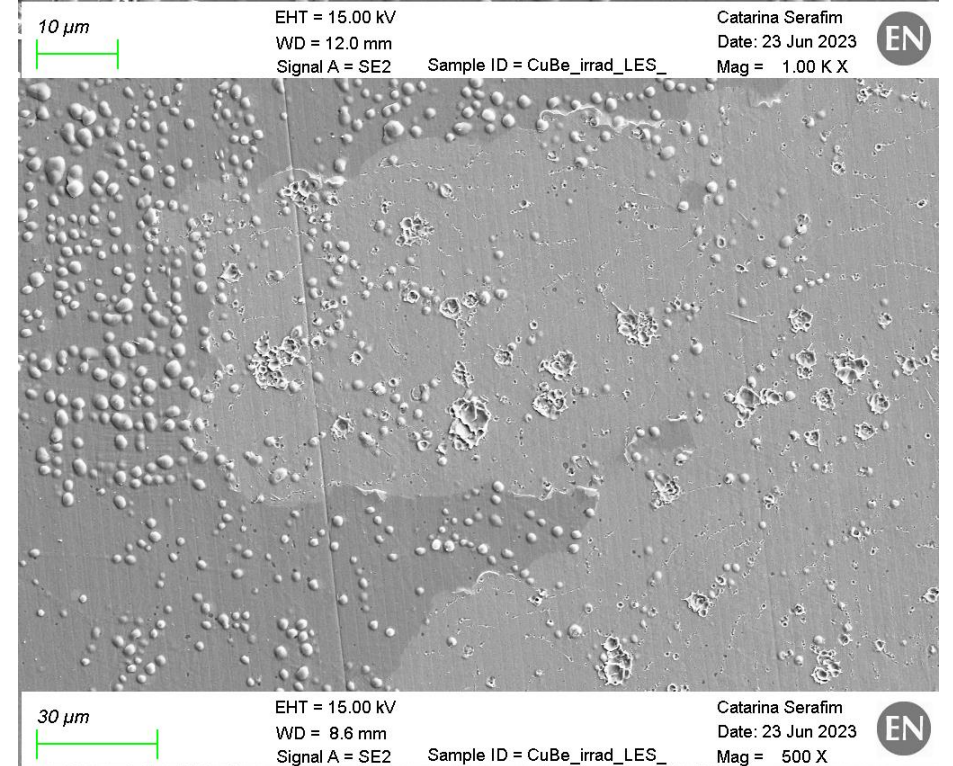
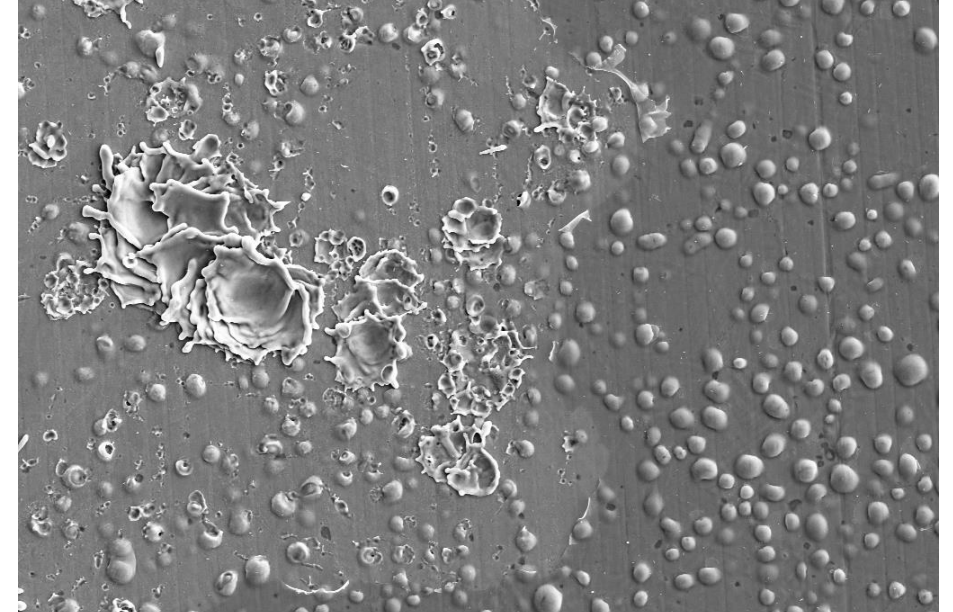
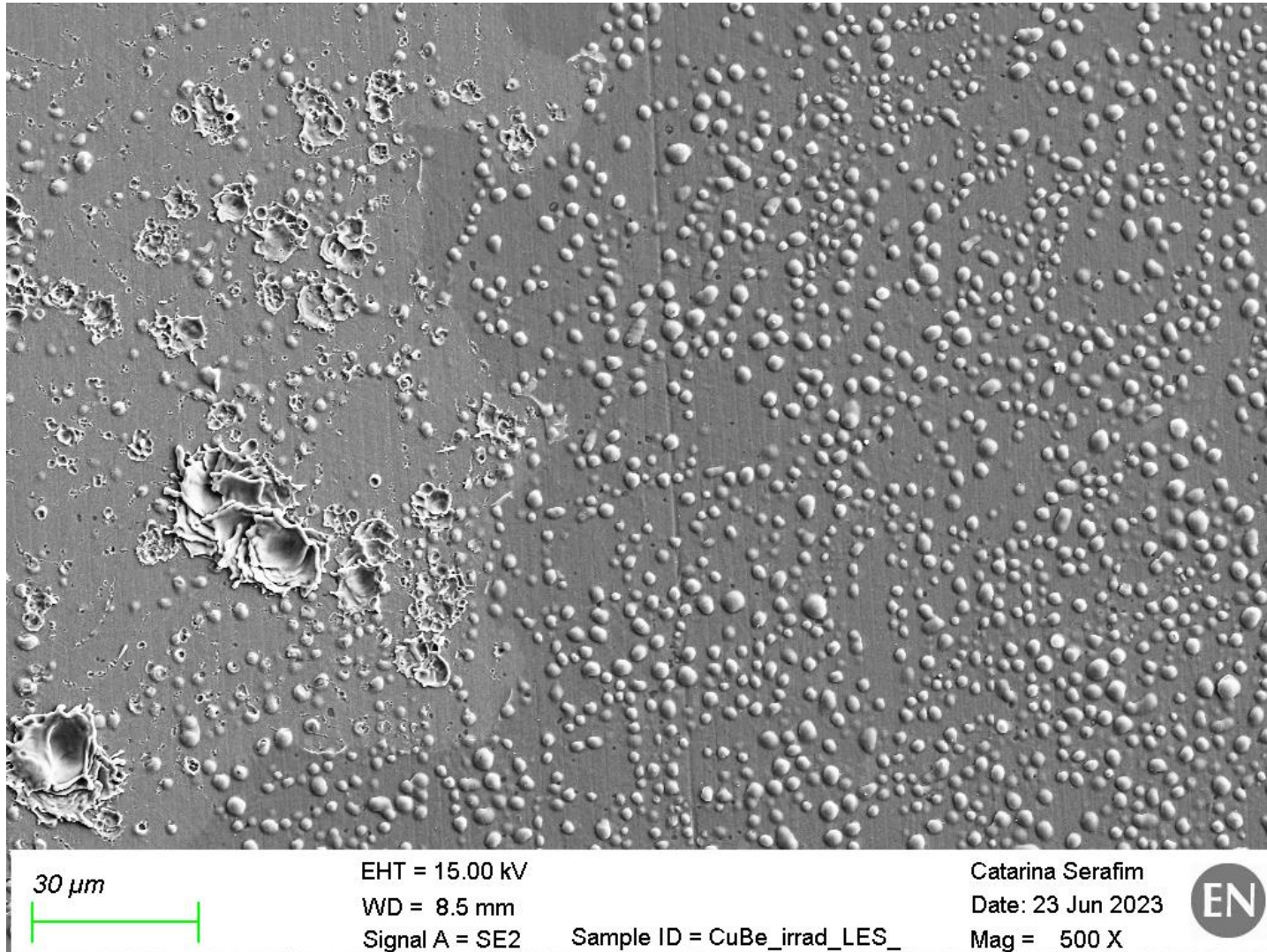
We have observed that the breakdowns are only concentrated inside the exfoliated zones.

The zones without those exfoliations, i.e. with a protective carbon layer, are free from breakdowns, even in the zones with high density of blisters.



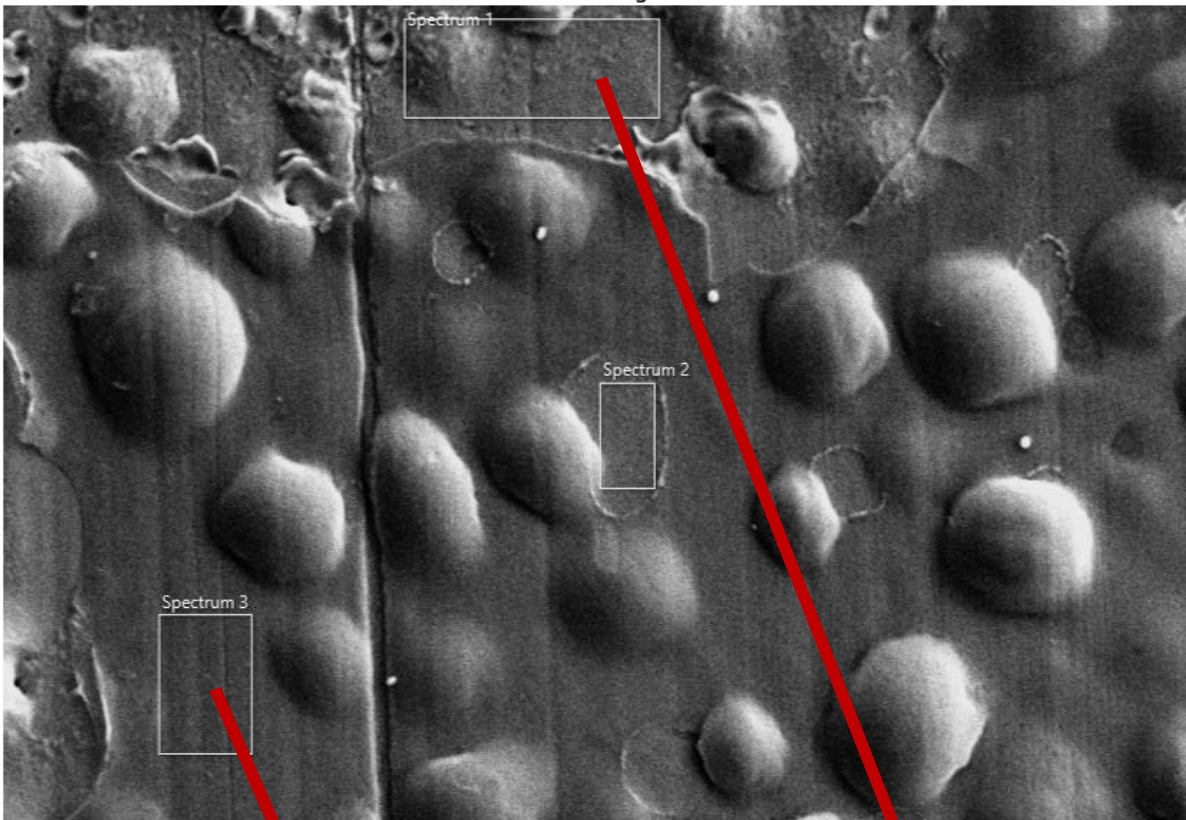


Zone with a higher density of blisters.  
The blisters have no influence on the triggering of breakdowns.



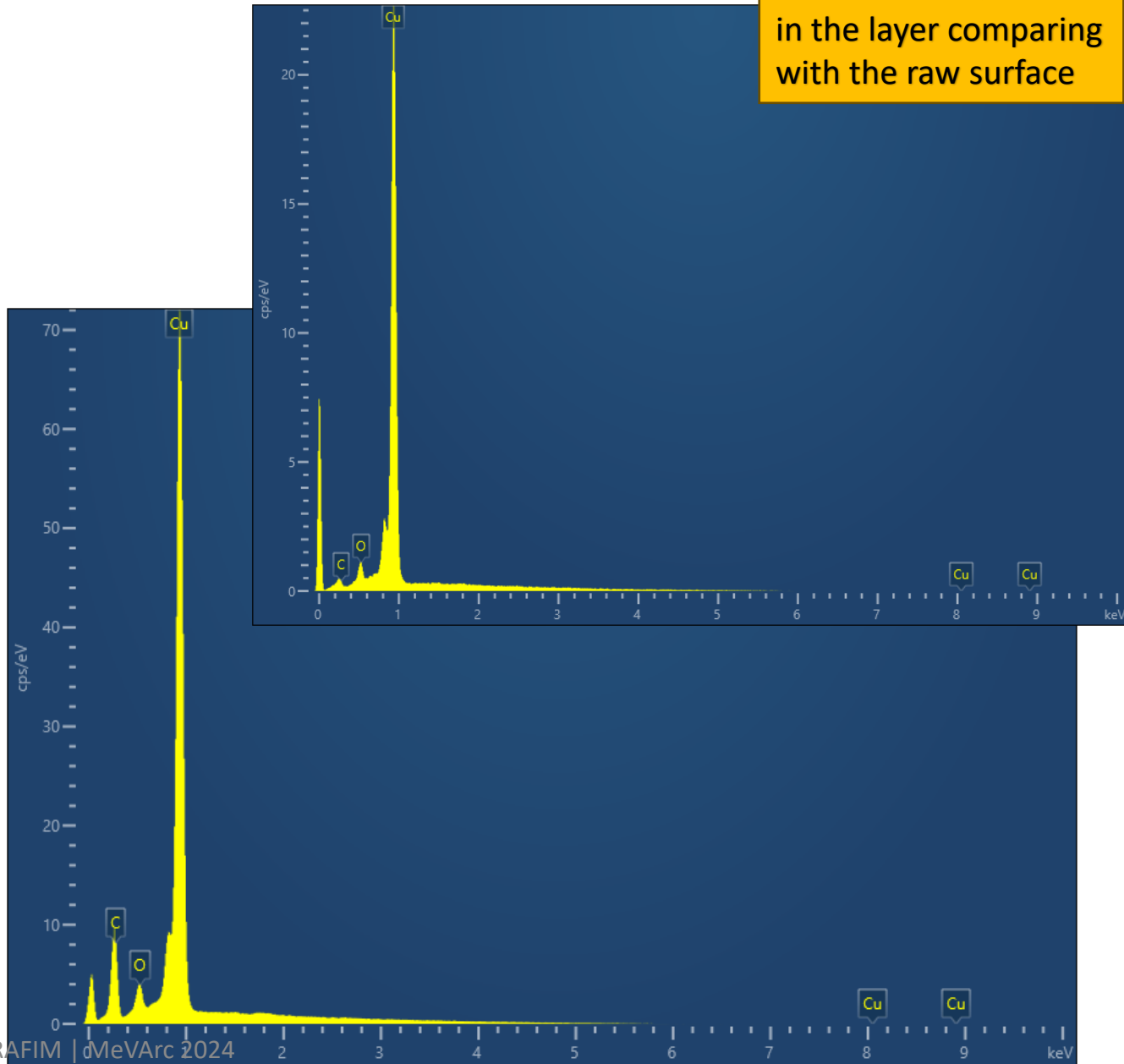


Electron Image 3



Element	Wt%
C	12.23
O	1.1
Cu	86.68

Element	Wt%
C	0.49
O	0.53
Cu	98.99

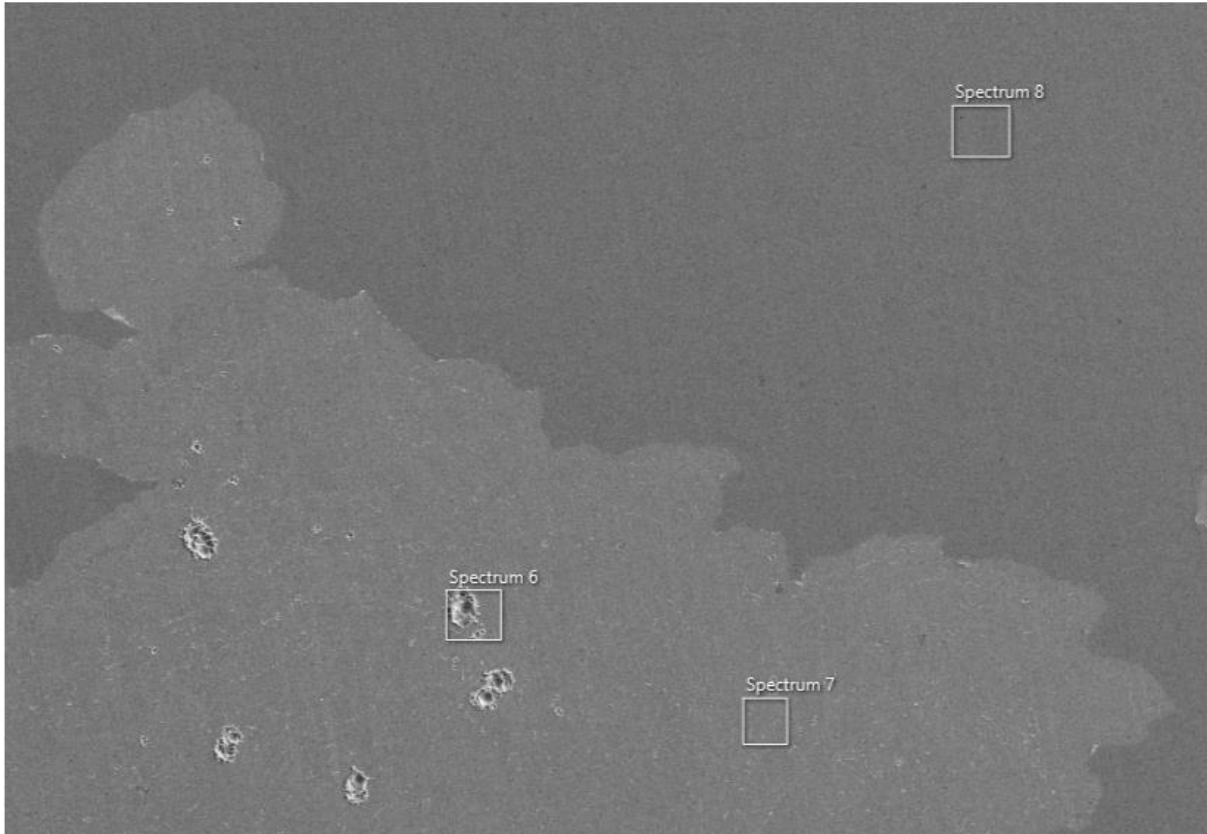


**Chemical analysis**  
More than 12% of C in  
in the layer comparing  
with the raw surface

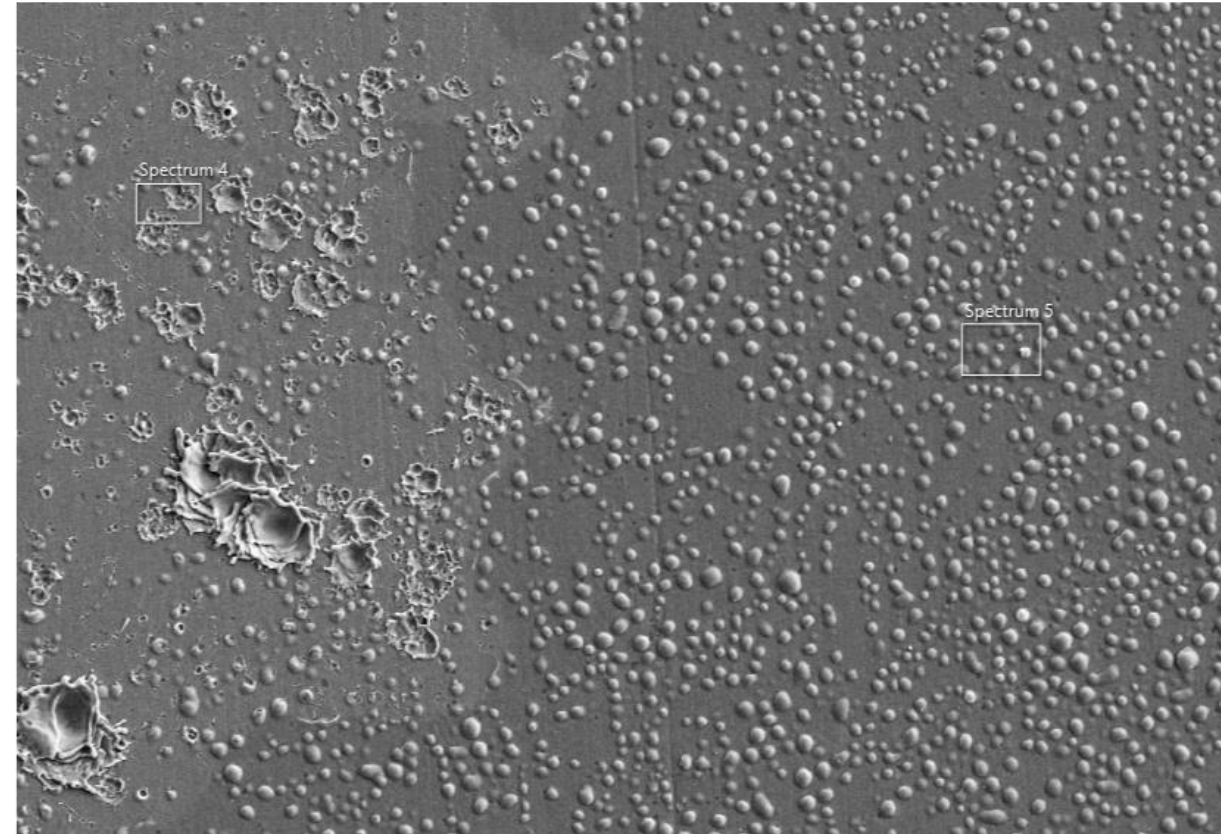


# Chemical analysis

Electron Image 4



Electron Image 2



**Spectrum 6**

Element	Line Type	Wt%
C	K series	1.25
Cu	L series	98.75

**Spectrum 7**

Element	Line Type	Wt%
C	K series	1.15
Cu	L series	98.85

**Spectrum 8**

Element	Line Type	Wt%
C	K series	8.86
Cu	L series	91.14

**Spectrum 4**

Element	Line Type	Wt%
C	K series	1.38
Cu	L series	98.62

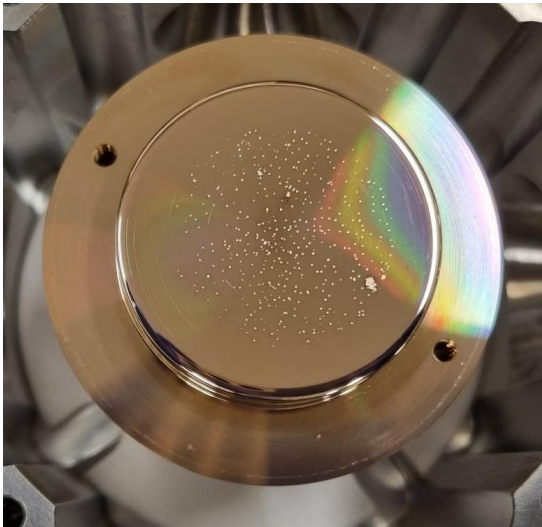
**Spectrum 5**

Element	Line Type	Wt%
C	K series	11.04
Cu	L series	88.96

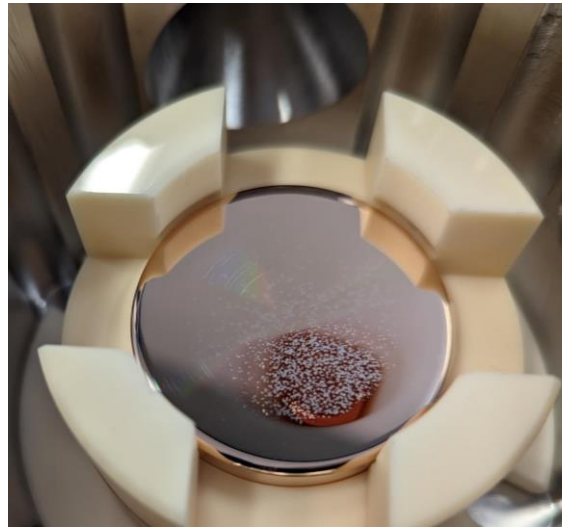


## The CuBe2 electrodes

CuBe2 non-irradiated  
achieved 90 MeV/m



CuBe2 irradiated (without  
plasma cleaning)  
achieved 45 MeV/m



CuBe2 irradiated  
(with plasma cleaning)

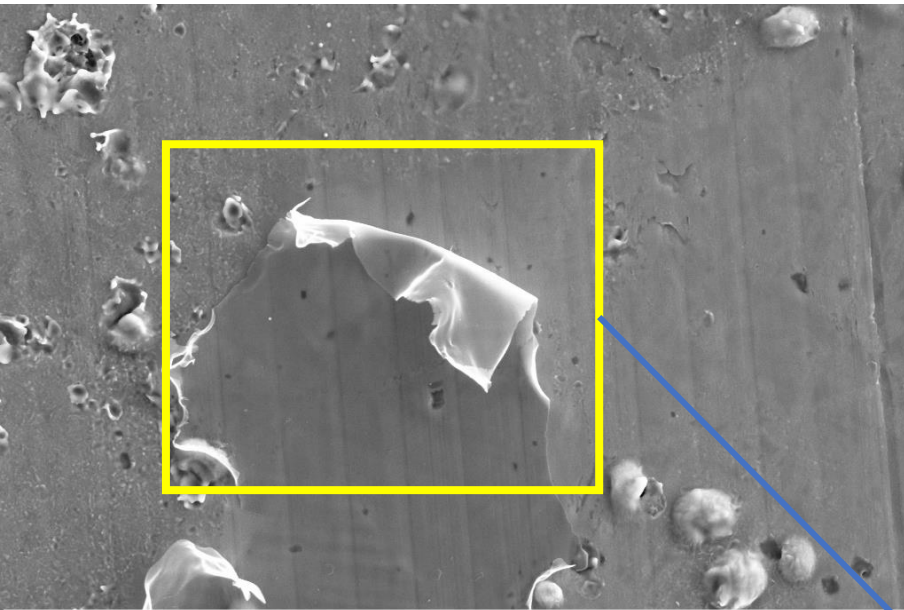


➔ Repeat LES testing after Plasma Treatment



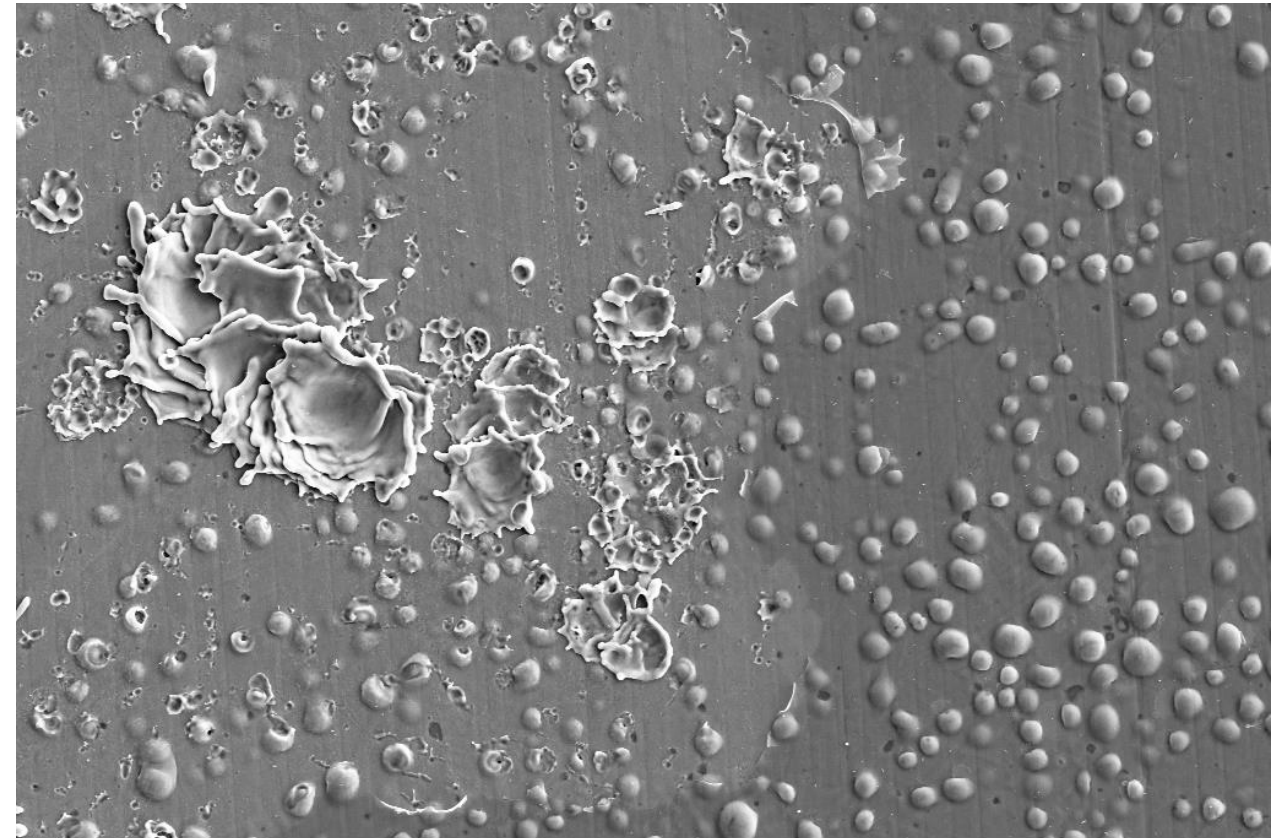
Is the carbon preventing the electrodes from reaching a high field?

Plan for Plasma cleaning treatment  
+ Repeat LES testing

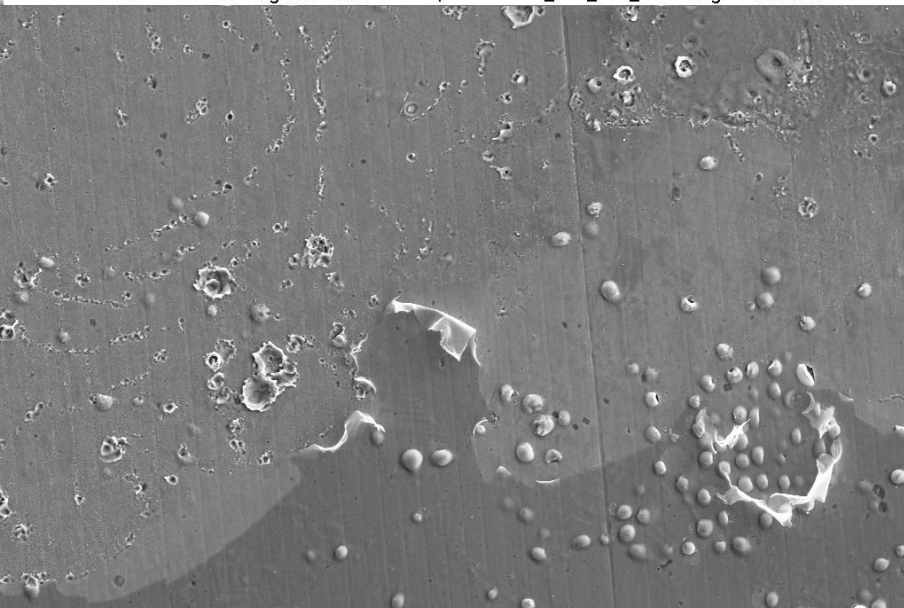


5  $\mu\text{m}$  EHT = 15.00 kV  
WD = 12.0 mm  
Signal A = SE2 Sample ID = CuBe\_irrad\_LES\_ Catarina Serafim  
Date: 23 Jun 2023  
Mag = 3.00 K X

Carbon layer

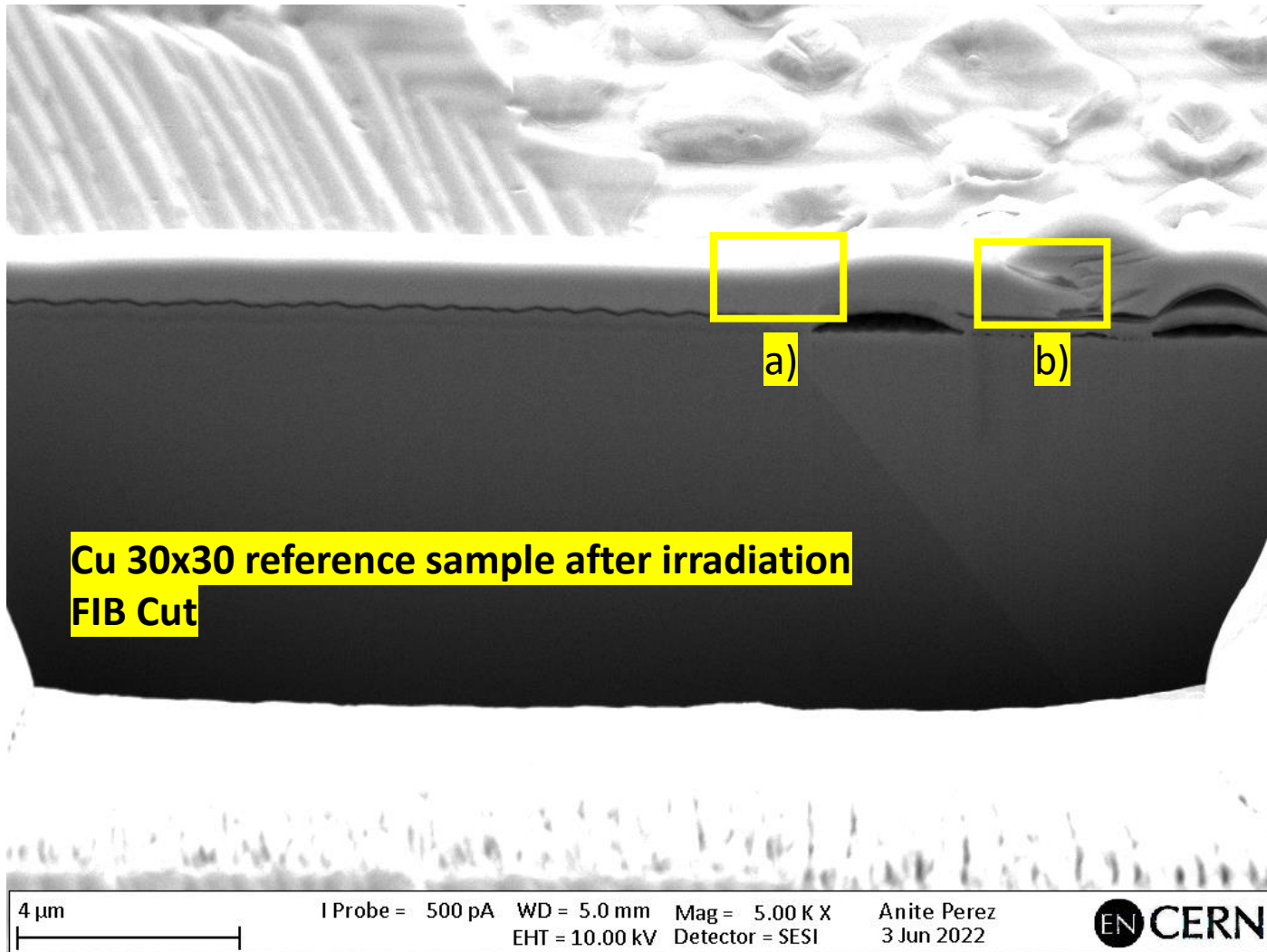


10  $\mu\text{m}$  EHT = 15.00 kV  
WD = 12.0 mm  
Signal A = SE2 Sample ID = CuBe\_irrad\_LES\_ Catarina Serafim  
Date: 23 Jun 2023  
Mag = 1.00 K X



10  $\mu\text{m}$  EHT = 15.00 kV  
WD = 12.0 mm  
Signal A = SE2 Sample ID = CuBe\_irrad\_LES\_ Catarina Serafim  
Date: 23 Jun 2023  
Mag = 1.00 K X



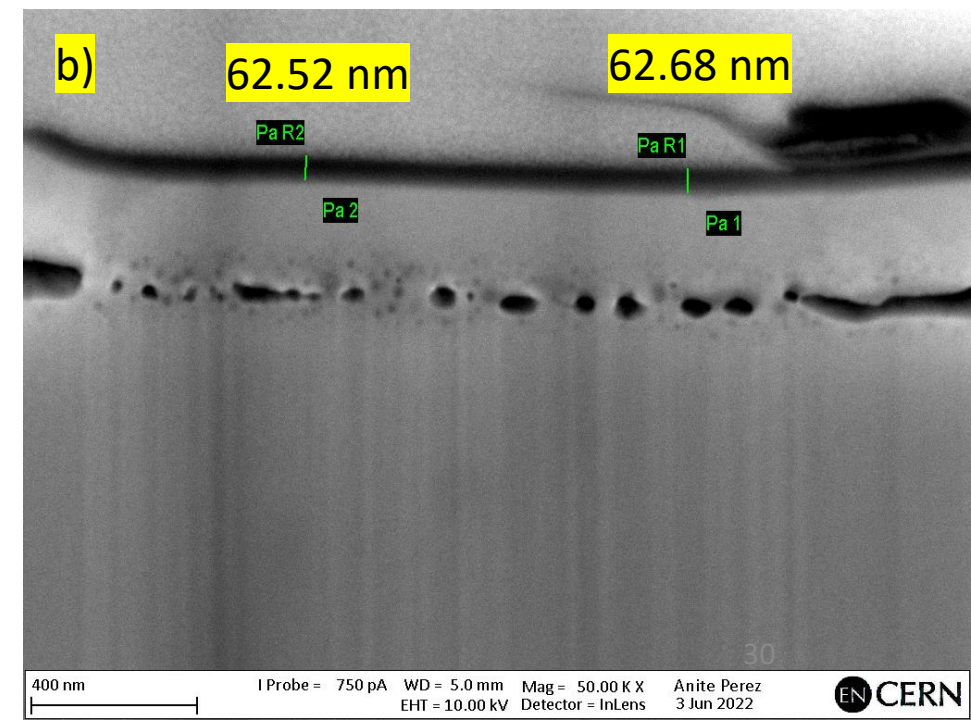
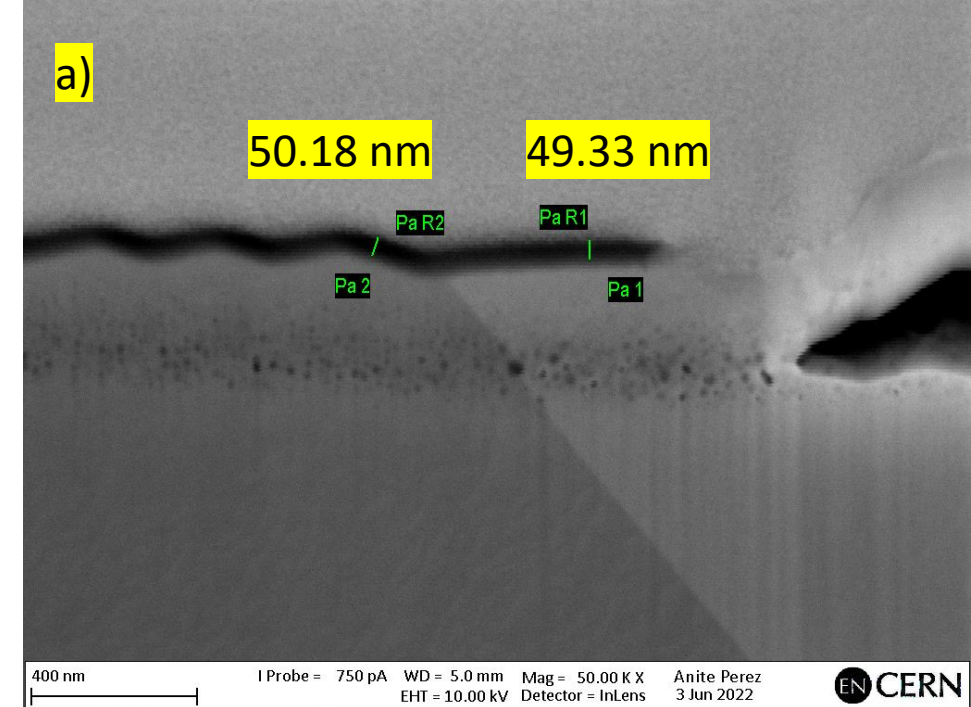


Data from a Cu sample previously analyzed after being exposed to irradiation (same parameters as the electrodes)

As we approach the irradiation zone we see an increase of the thickness of the C with a factor of approx. 2

Cu layer approx. 56 nm

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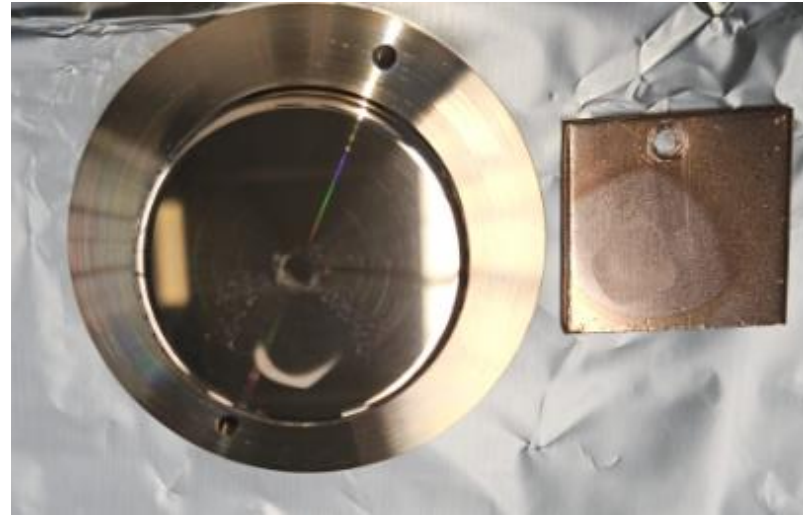
# Plasma cleaning of H- irradiated CuBe cathode and reference Cu sample irradiated



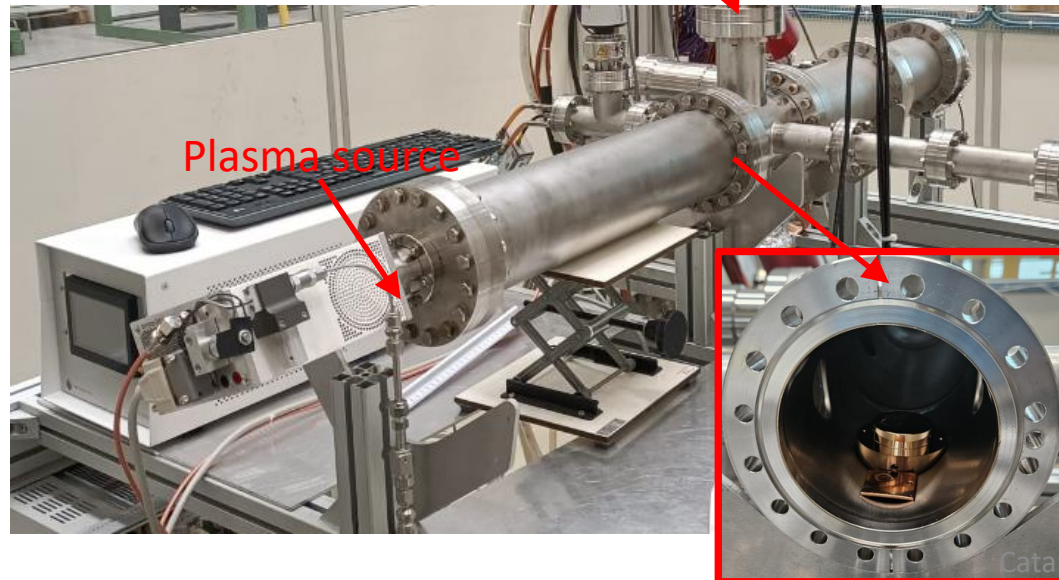
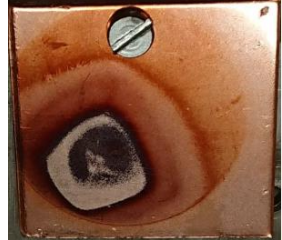
System in 181

viewport

After O<sub>2</sub> plasma cleaning



Before O<sub>2</sub> plasma cleaning



Plasma source

## Plasma treatment parameters

Plasma source: ibss alumina tube 300W prototype,  $\phi = 0.147$  in

$p = 4E-3$  mbar, pure oxygen (pressure)

$P_{rf} = 50$  W (pl.source rf power)

$L = 610 \text{ mm} \pm 10 \text{ mm}$  (distance samples - plasma source);  $D_{ch} = 100 \text{ mm}$

a-C dth/dt in these conditions = 0.015 nm/s (removal rate)

Plasma ion energy in these conditions : 35 eV (measured by RFEA)

Treatment duration = 1h 15 min (nominally 68 nm of a-C)



After 3 hours of treatment in total

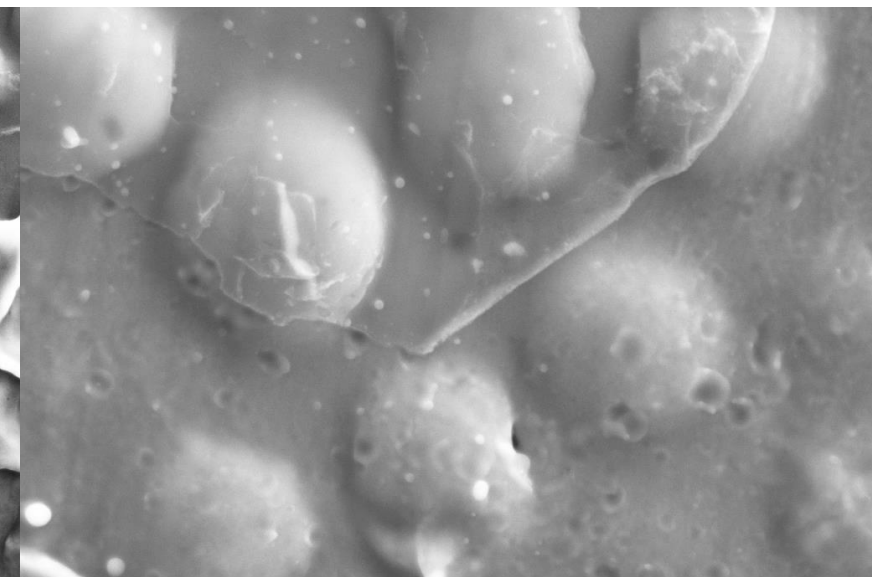
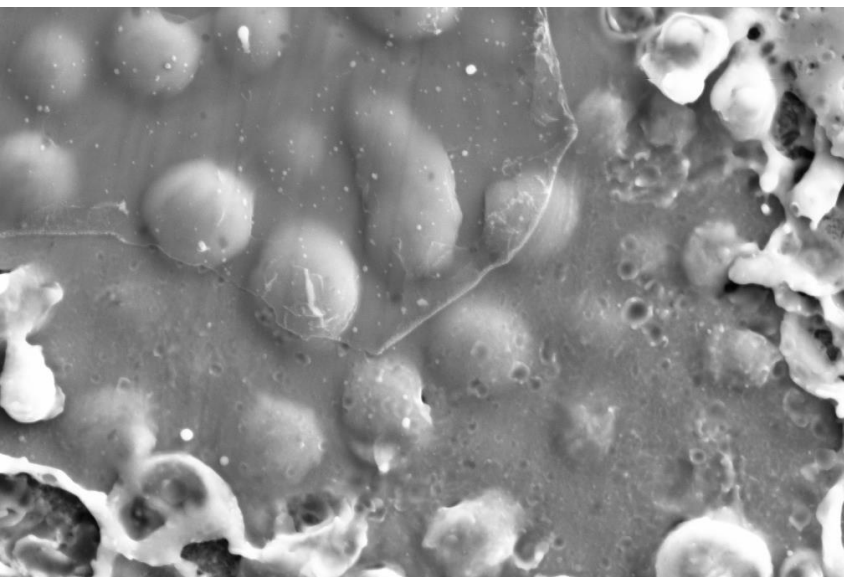
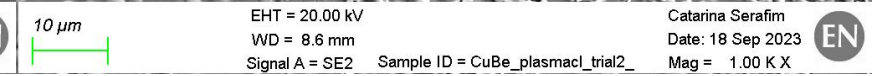
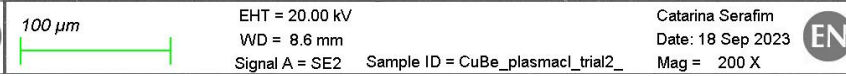
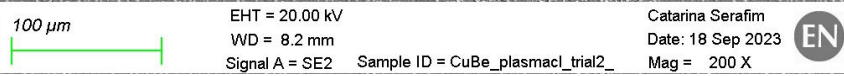
Seems that the carbon layer on the surface has significant smaller thickness.

From SEM we see this layer being more transparent.

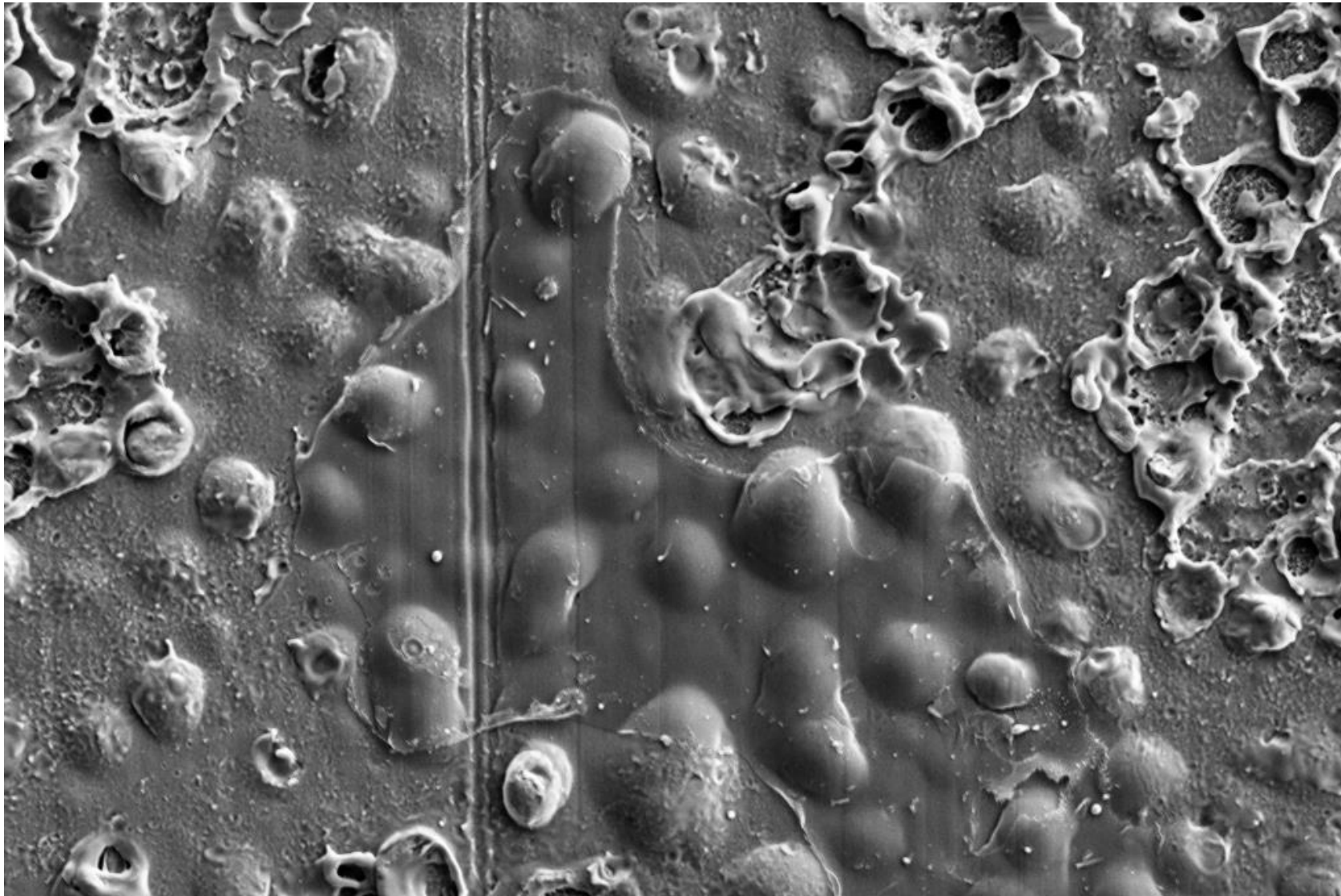
Big discoloration between regions with and without carbon are not so evident as before.

Decrease by factor of 2 of the percentage of Carbon.

Between 4 -5 % of C. Other regions around 1%







5  $\mu$ m

EHT = 6.00 kV  
WD = 8.3 mm  
Sample ID = 3rdplasmacleaning\_cathode

Signal A = SE2

Catarina Serafim  
Date: 27 Sep 2023  
Mag = 3.00 K X



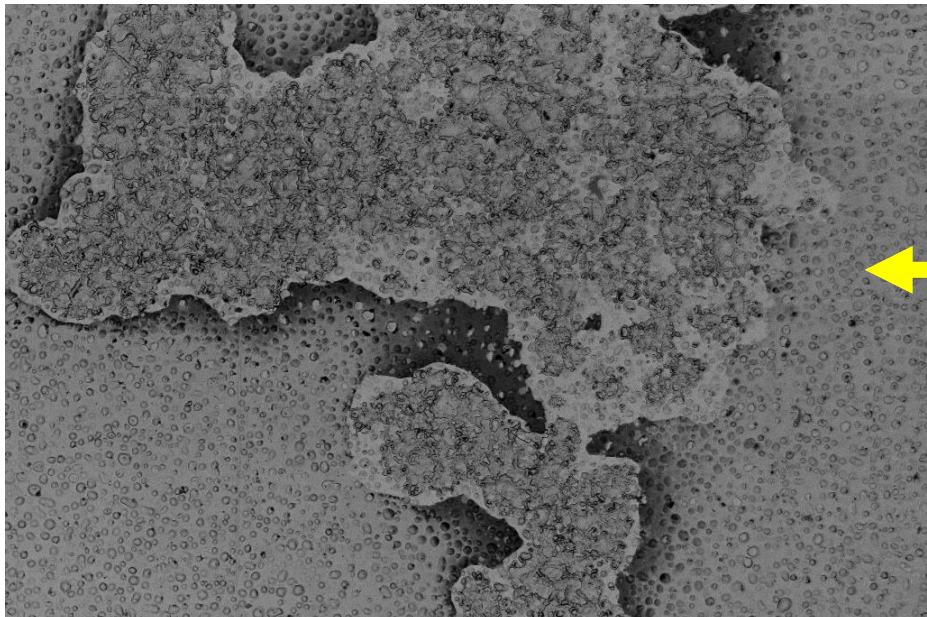
**In total 6h hours** of treatment was preformed.

The quantity of C in the surface is now significantly lower.

We have concluded that because of the roughness of the surface (due to breakdowns from the LES test), C seems to be very difficult to be removed in these regions (see next images).

For the other hand, the regions without breakdowns seem to be cleaned (1-2% of C).

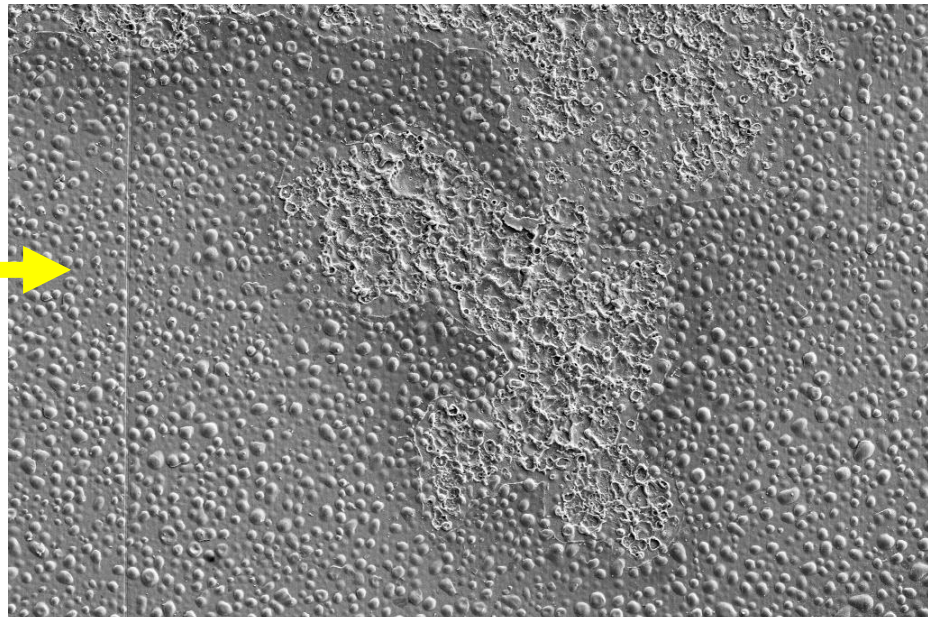




40 μm

EHT = 6.00 kV  
WD = 8.8 mm  
Signal A = AsB  
Sample ID = 3rdplasmacleaning\_cathode

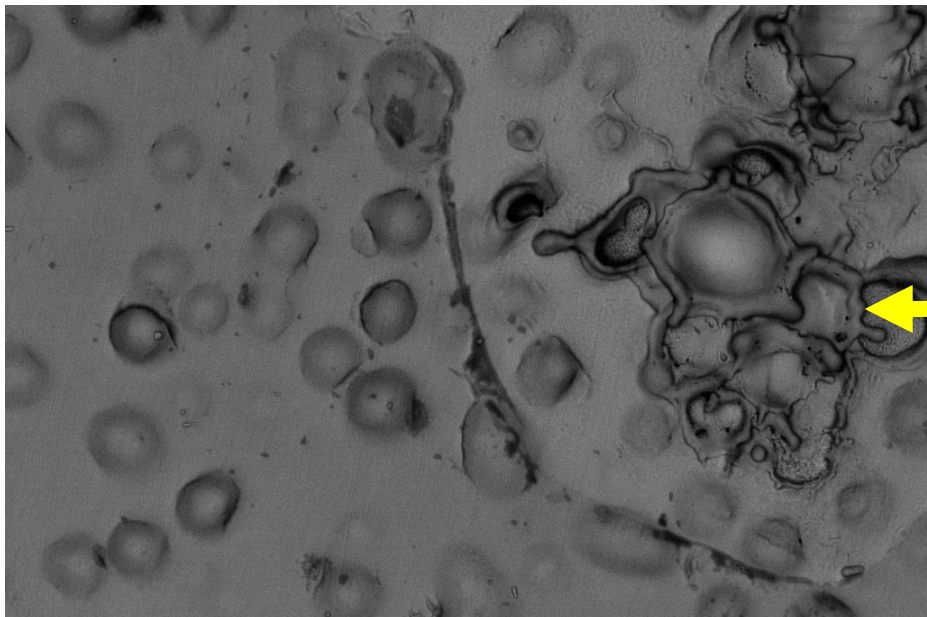
Catarina Serafim  
Date: 27 Sep 2023  
Mag = 372 X



30 μm

EHT = 6.00 kV  
WD = 8.8 mm  
Signal A = SE2  
Sample ID = 3rdplasmacleaning\_cathode

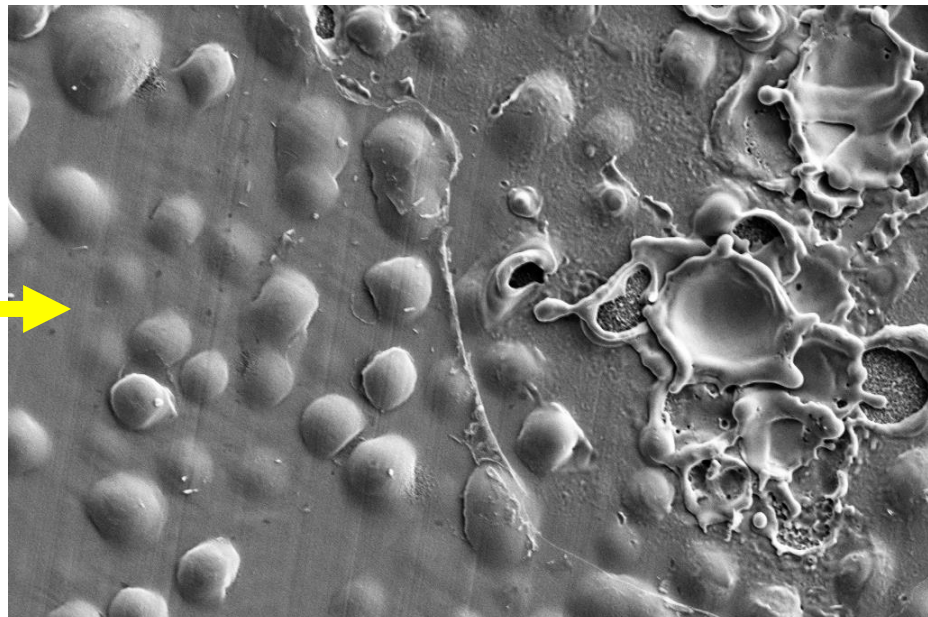
Catarina Serafim  
Date: 27 Sep 2023  
Mag = 500 X



5 μm

EHT = 6.00 kV  
WD = 8.8 mm  
Signal A = AsB  
Sample ID = 3rdplasmacleaning\_cathode

Catarina Serafim  
Date: 27 Sep 2023  
Mag = 3.00 K X



5 μm

EHT = 6.00 kV  
WD = 8.8 mm  
Signal A = SE2  
Sample ID = 3rdplasmacleaning\_cathode

Catarina Serafim  
Date: 27 Sep 2023  
Mag = 3.00 K X

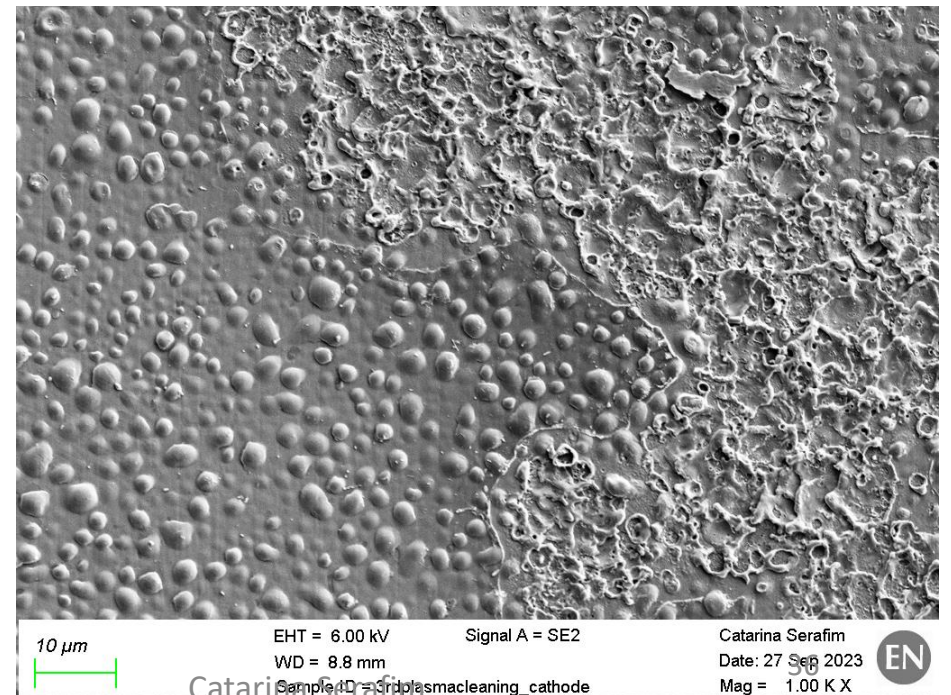
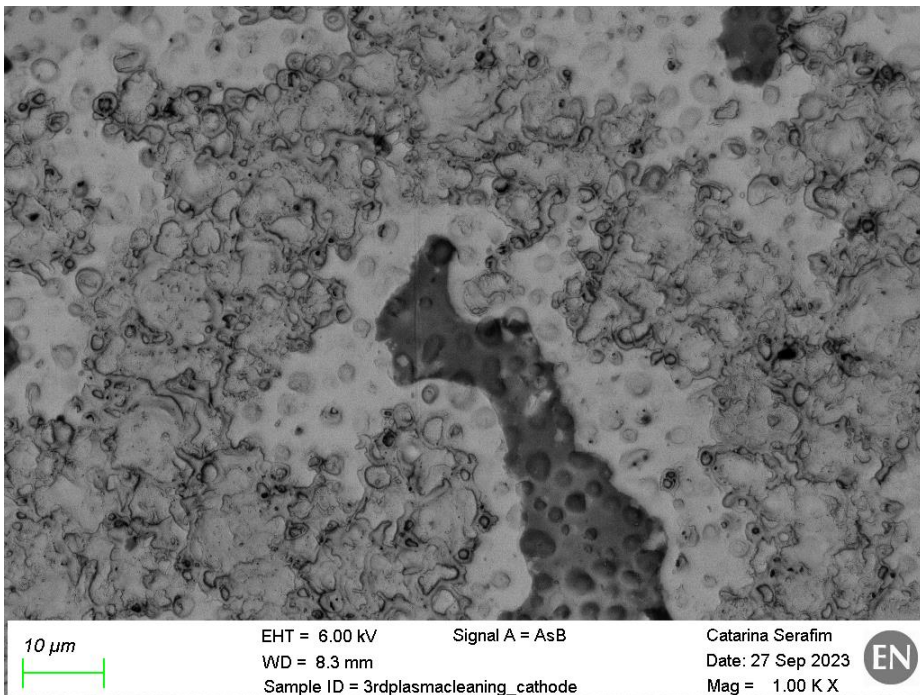
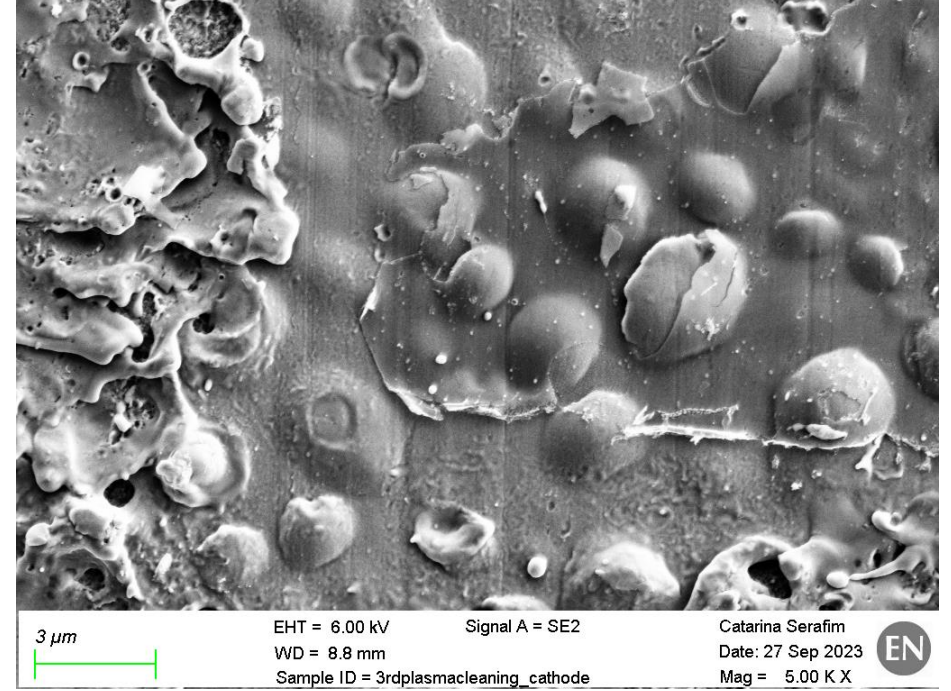
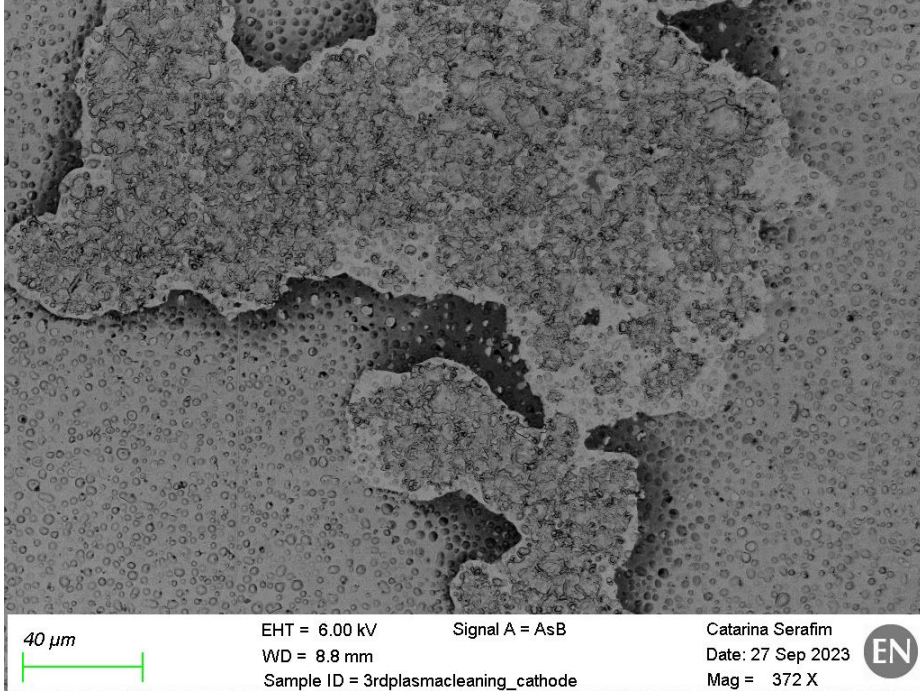


Backscattered detector

Secondary electron detector



Imaging using Backscattered detector. Darker color means the detection of lighter elements (in our case C).

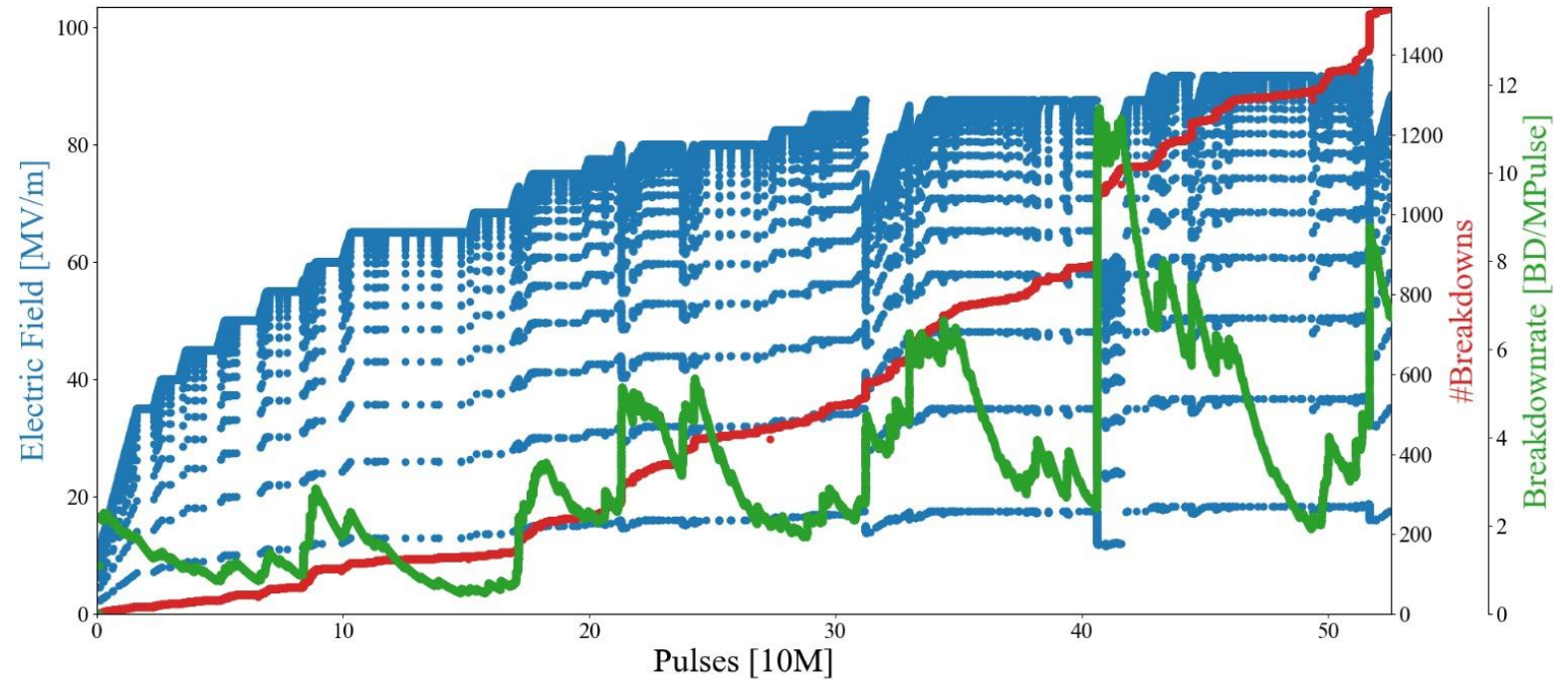
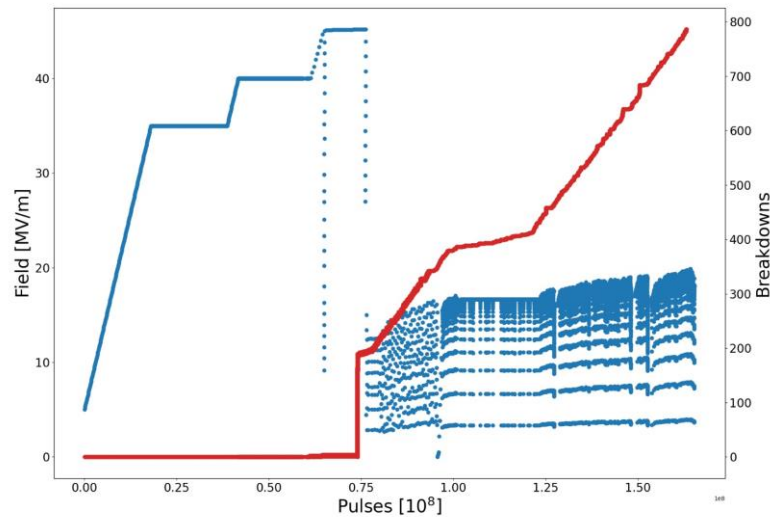




# CuBe2 breakdown test

The followings plots are from the same set of electrodes:

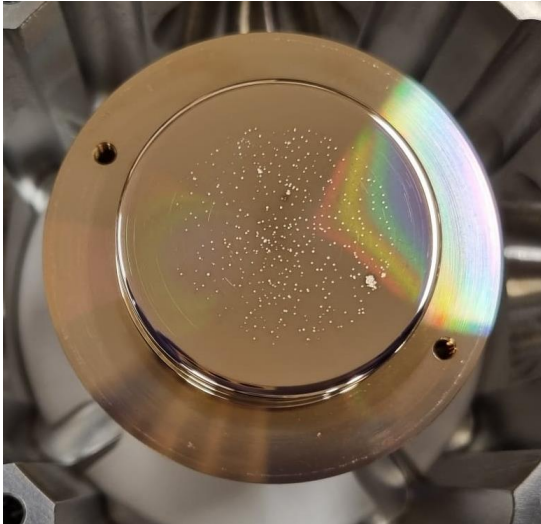
- On the **left** before plasma treatment
- On the **right** after plasma treatment



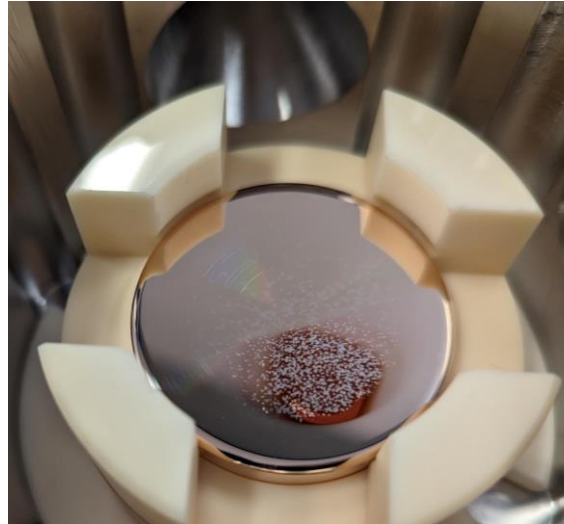


After the plasma cleaning, we decided to re-test the electrodes in the LES system

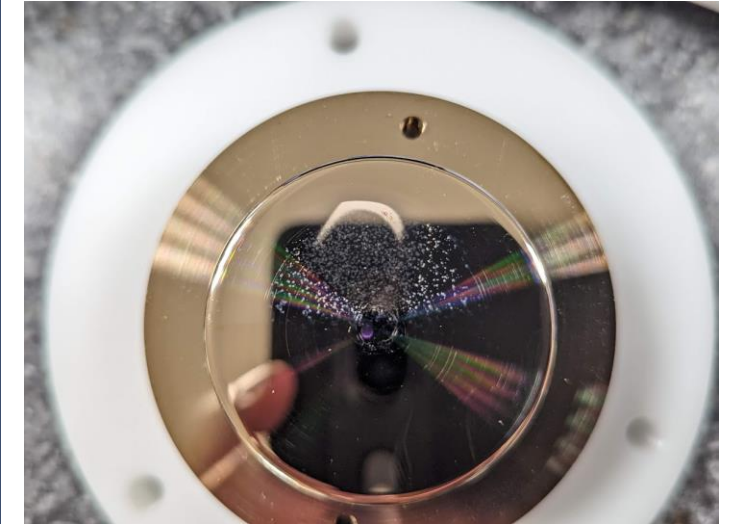
CuBe2 non-irradiated  
achieved 90 MeV/m



CuBe2 irradiated (without  
plasma cleaning)  
achieved 45 MeV/m



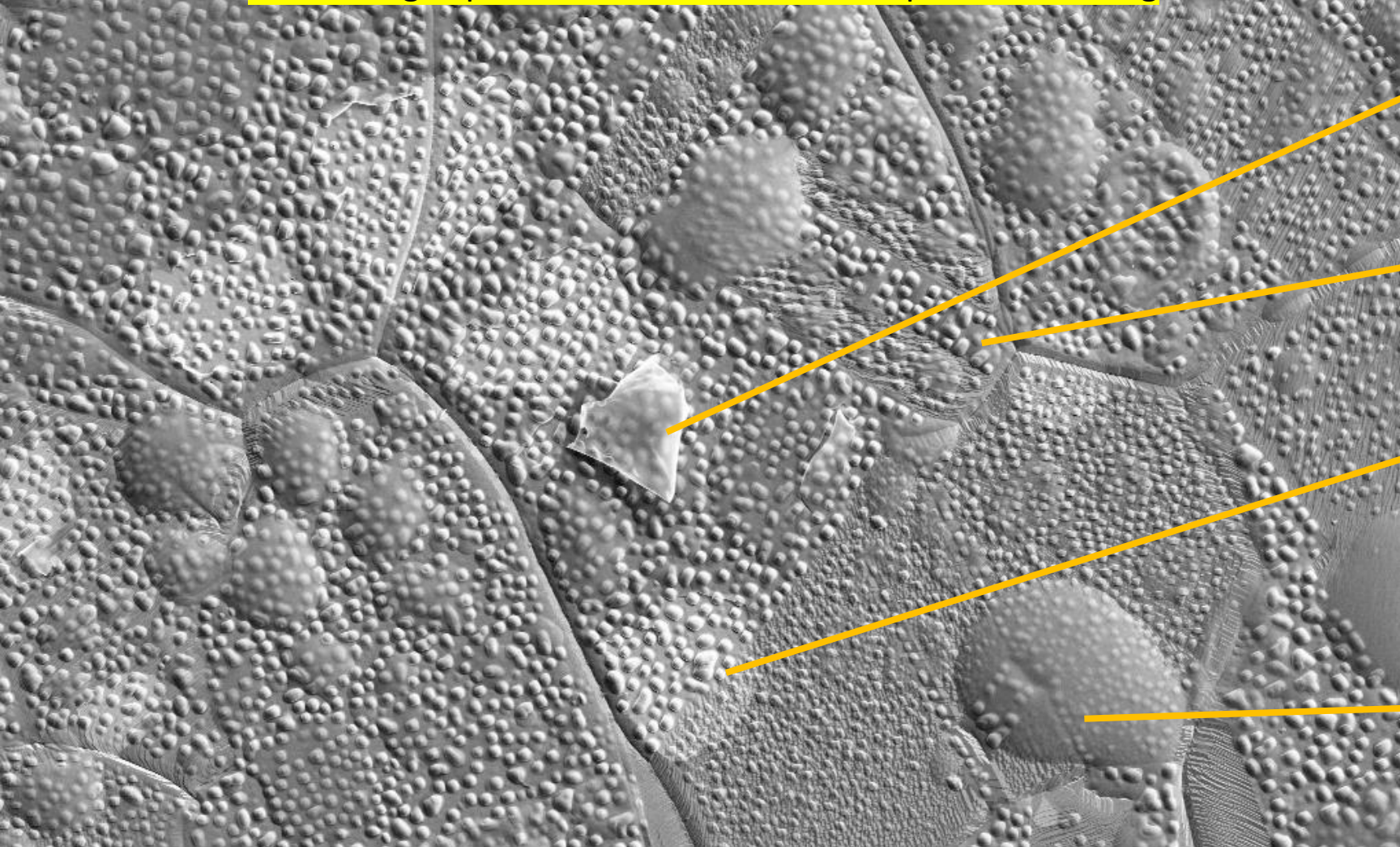
CuBe2 irradiated  
achieved 82 MeV/m (with  
plasma cleaning)



- We have concluded that the plasma treatment was a success for Carbon removal from the surface. The electrode for the trial was CuBe2. This electrode has been exposed to irradiation and also has been tested with high pulsing voltage in a dedicated system (LES), which has triggered the breakdowns on the surface.
- The next step was to perform the treatment also in two different electrodes (Cu-OFE and Stainless Steel) right after the irradiation (without high voltage tests). We hope to eliminate completely the C residuals that we still see on CuBe after 6h hours of treatment.



SEM Images (secondary electron detector) of Cu-OFE electrode after being exposed with H- beam before plasma cleaning



Carbon layer that was partially exfoliated from the surface

Blisters caused by the irradiation with H- beam

Regions without carbon (white regions). C has been completely exfoliated

'Bubbles' of C – implementation of H between C layer and Cu surface

30  $\mu$ m

EHT = 5.00 kV

Signal A = SE2

Catarina Serafim

WD = 8.2 mm

Date: 31 Oct 2023

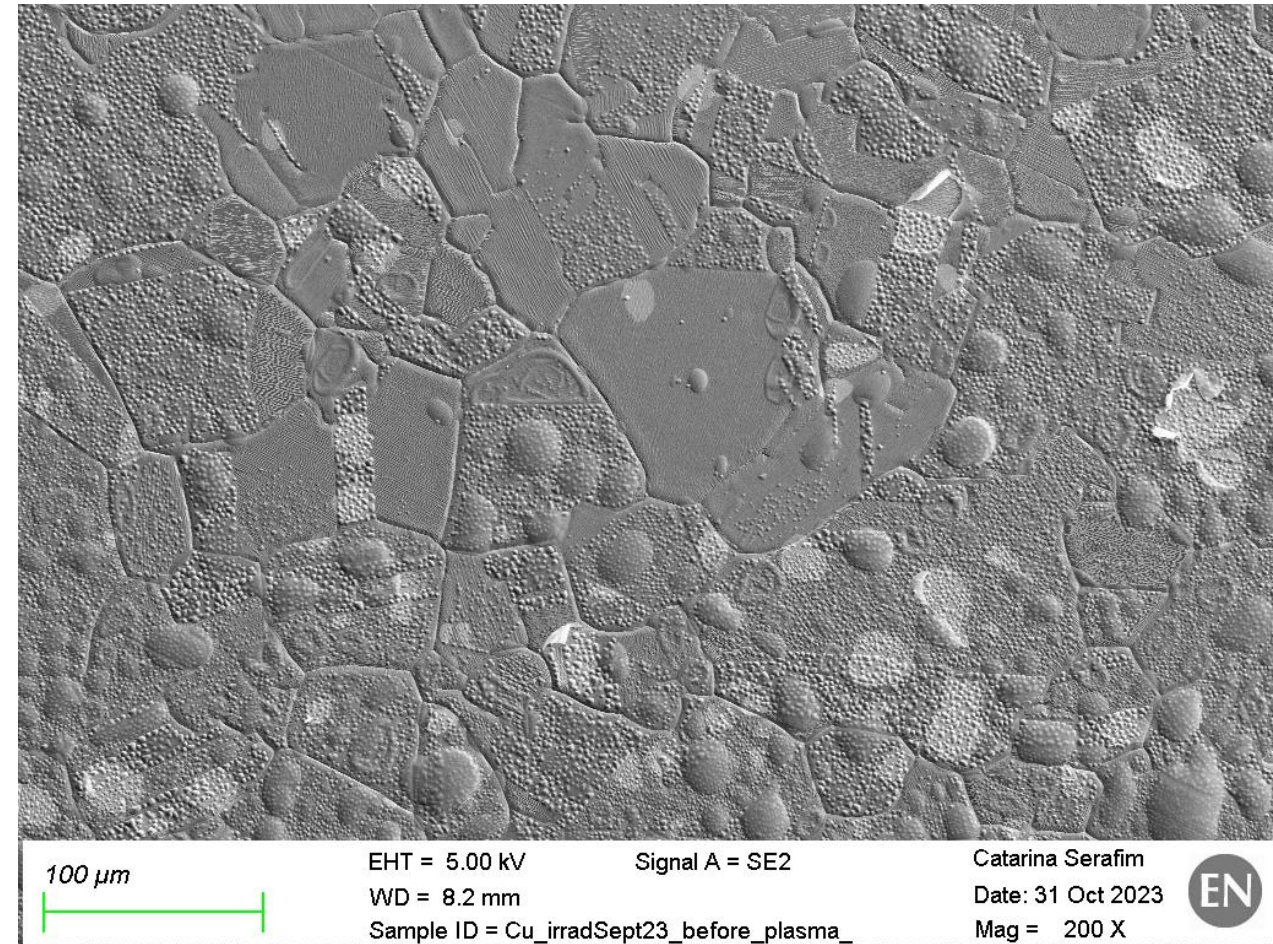
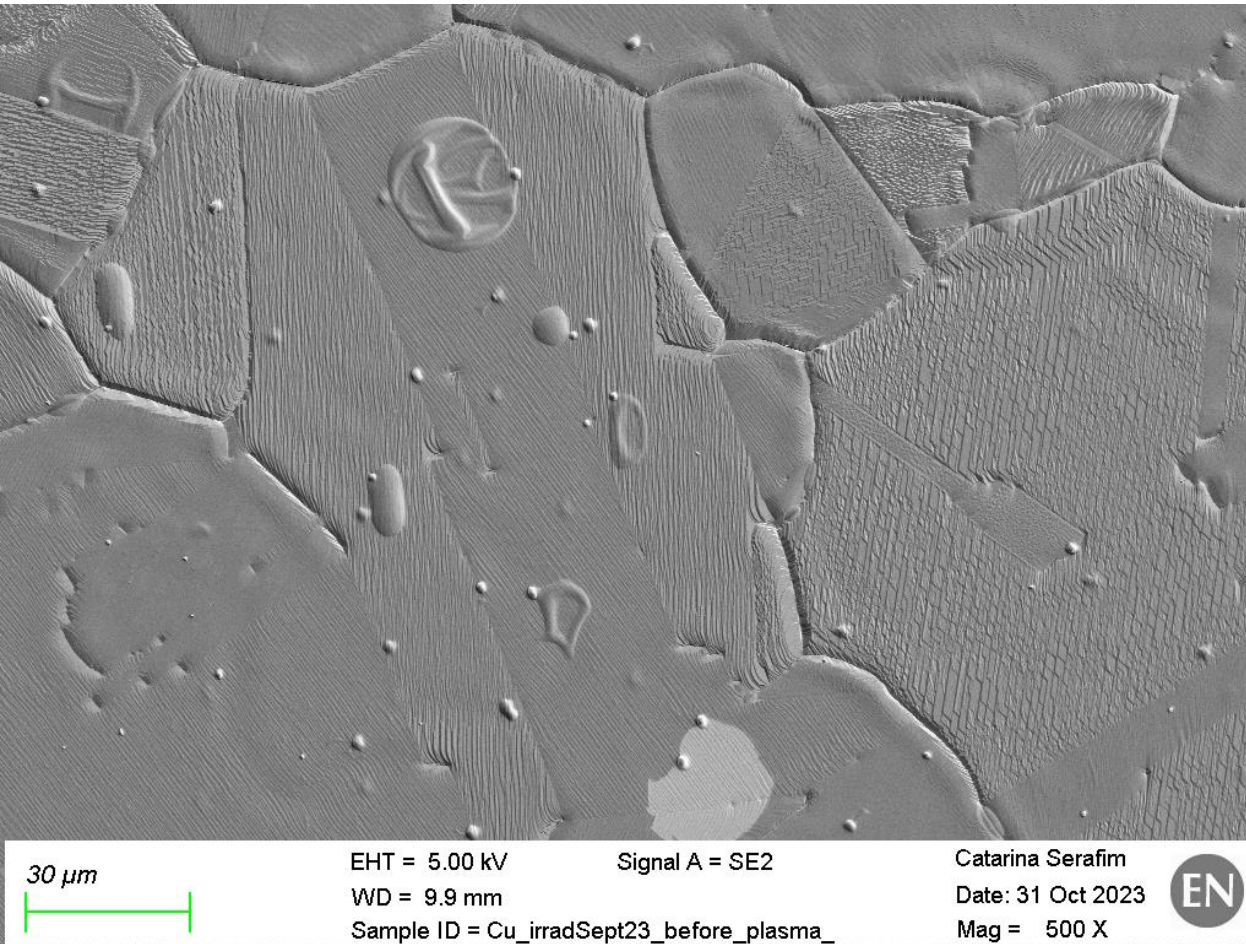
Sample ID = Cu\_irradSept23\_before\_plasma\_

Mag = 500 X



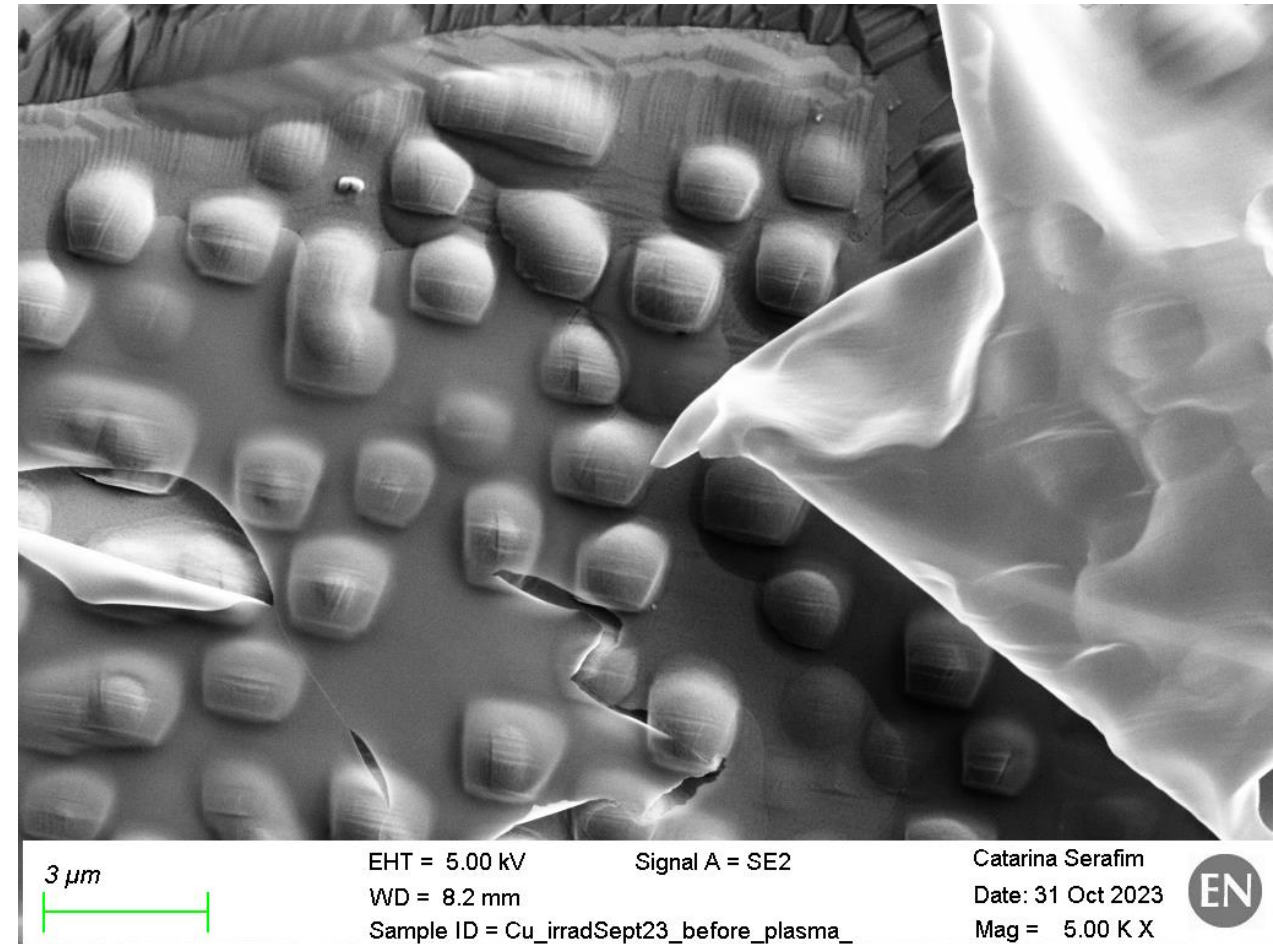
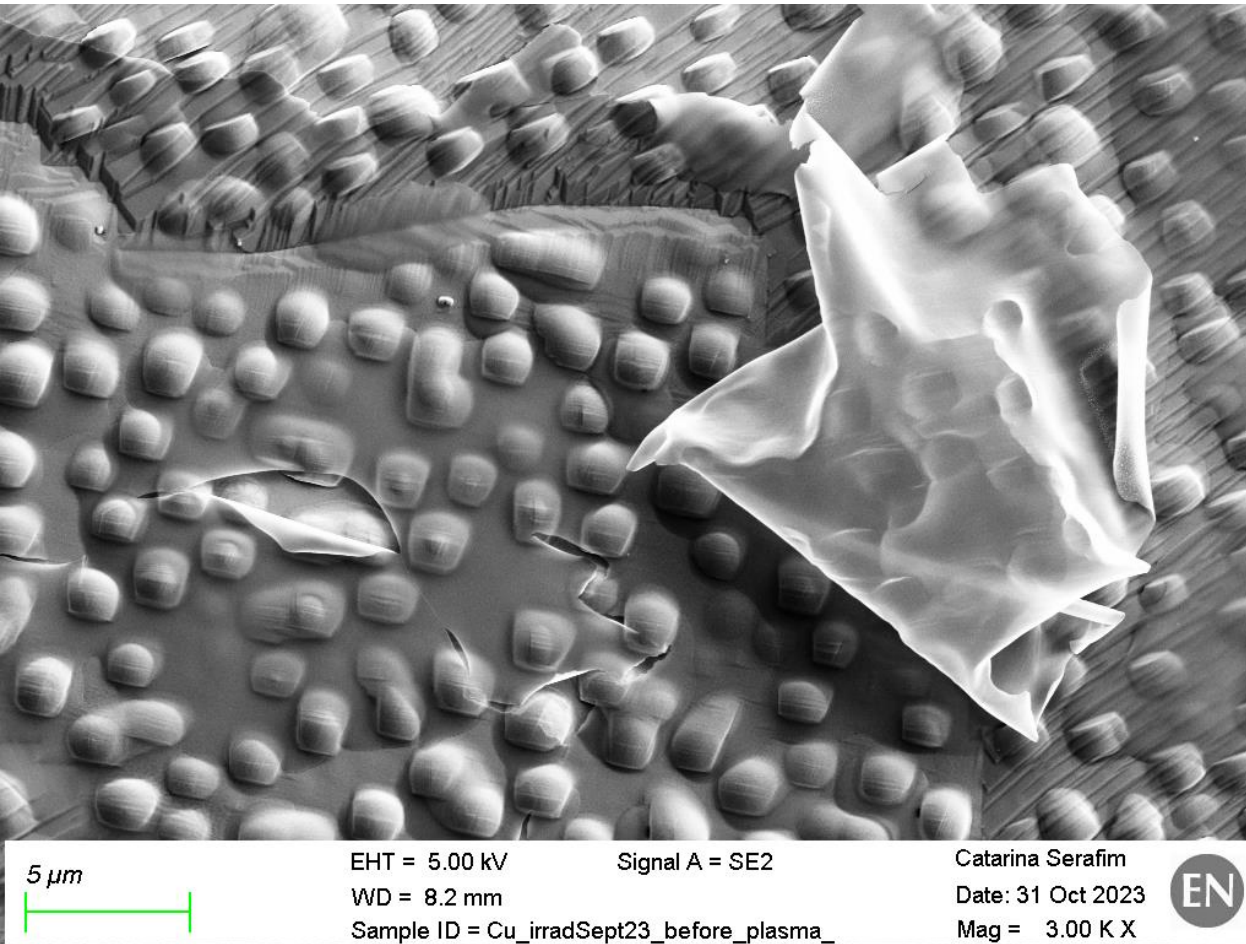


SEM Images (secondary electron detector) of Cu-OFE electrode  
after being exposed with H- beam before plasma cleaning



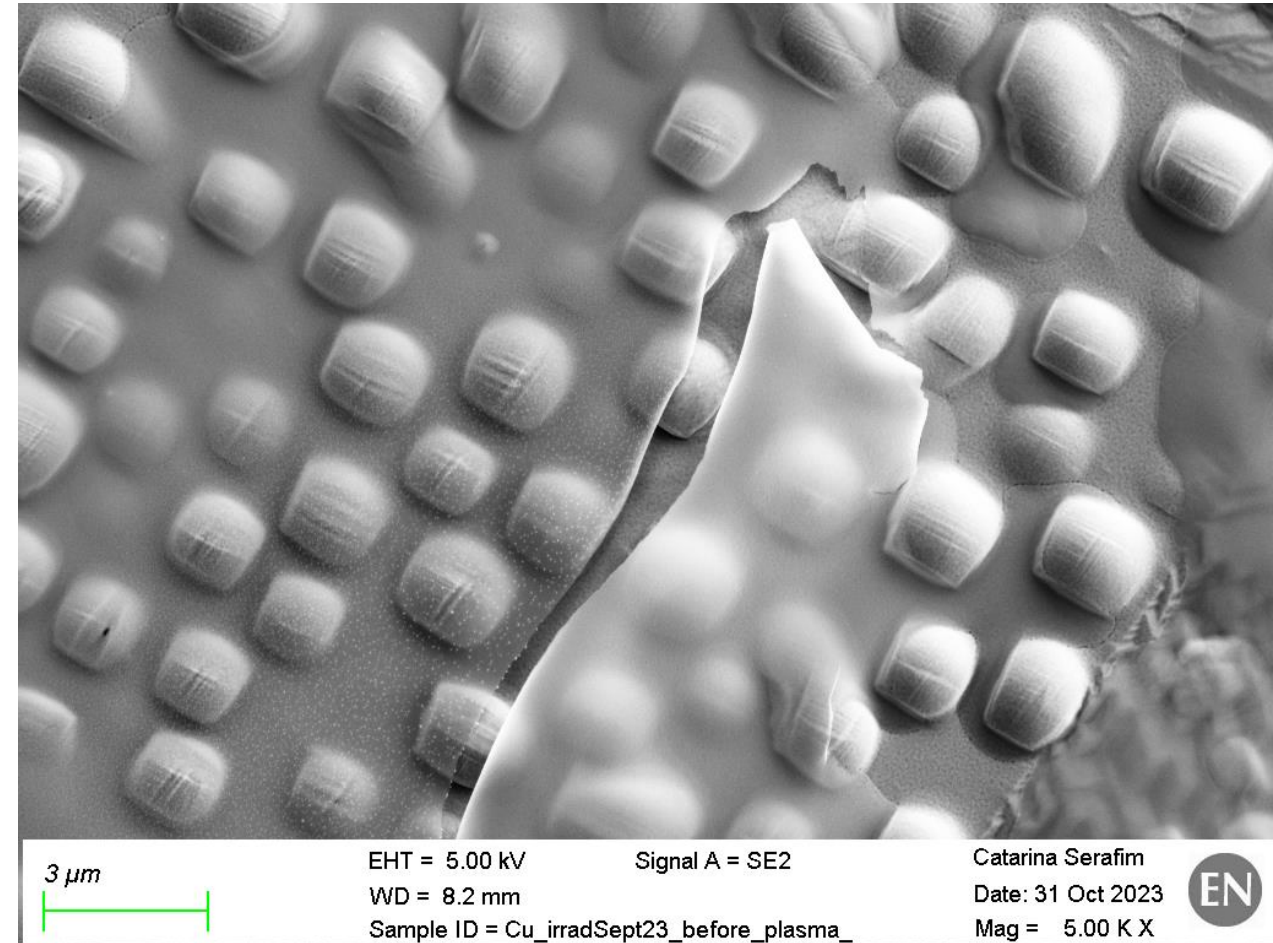
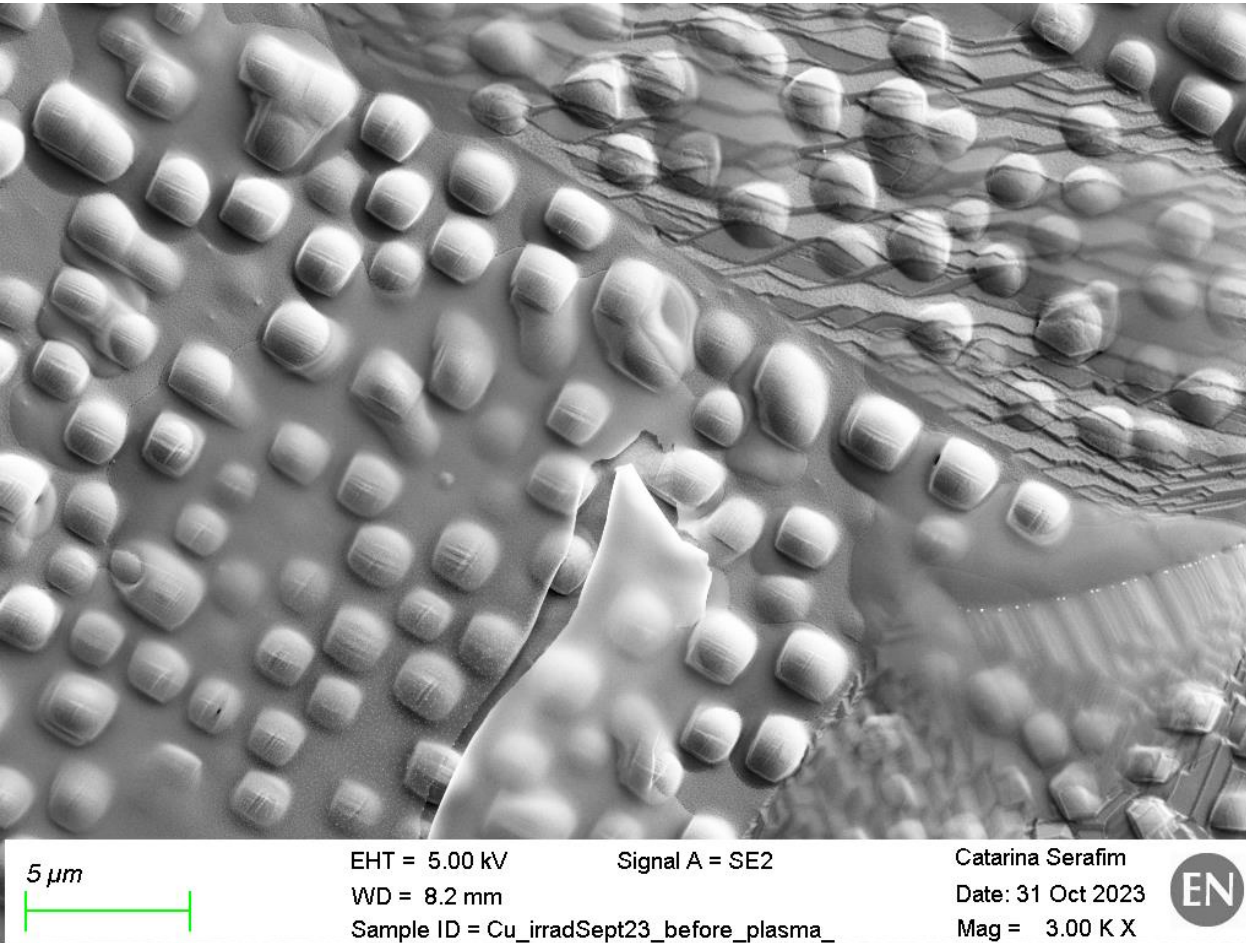


SEM Images (secondary electron detector) of Cu-OFE electrode  
after being exposed with H- beam before plasma cleaning



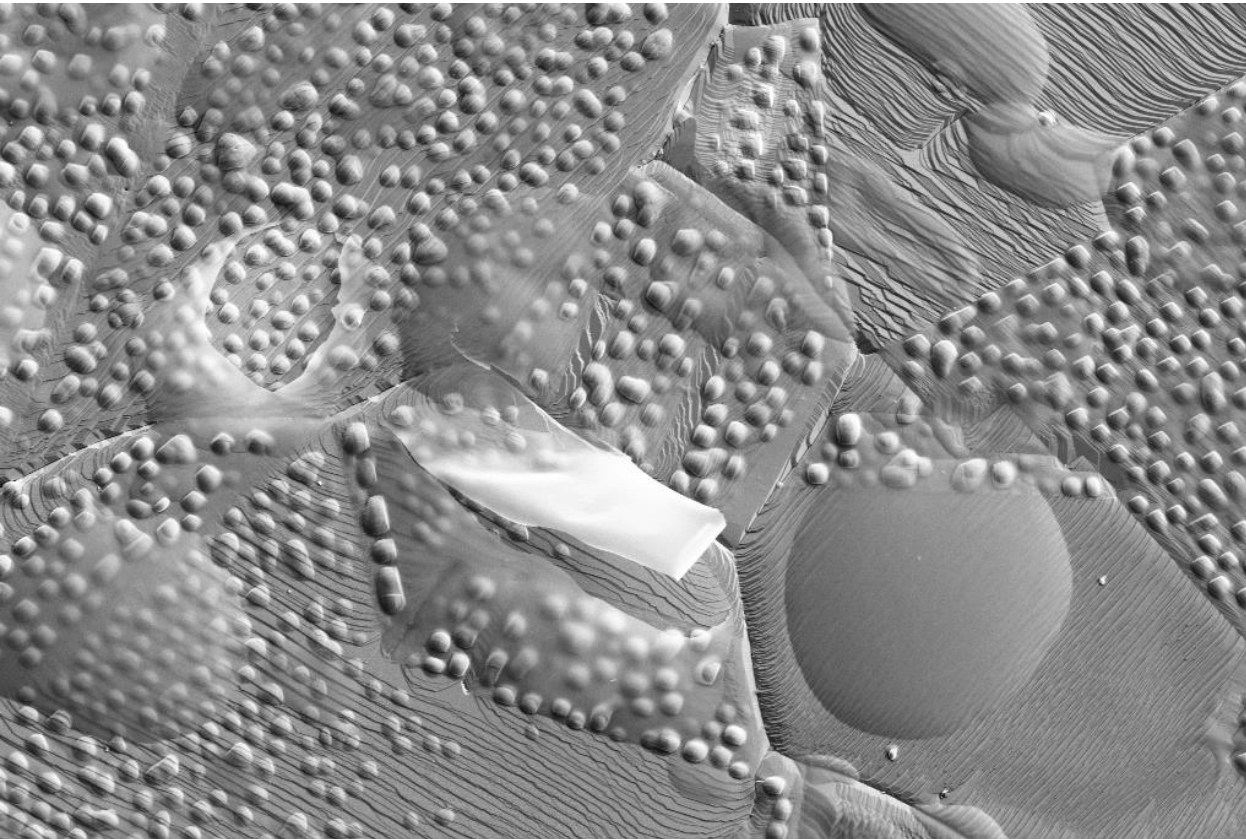


SEM Images (secondary electron detector) of Cu-OFE electrode  
after being exposed with H- beam before plasma cleaning





SEM Images (secondary electron detector) of Cu-OFE electrode  
after being exposed with H- beam before plasma cleaning



EHT = 5.00 kV

Signal A = SE2

Catarina Serafim

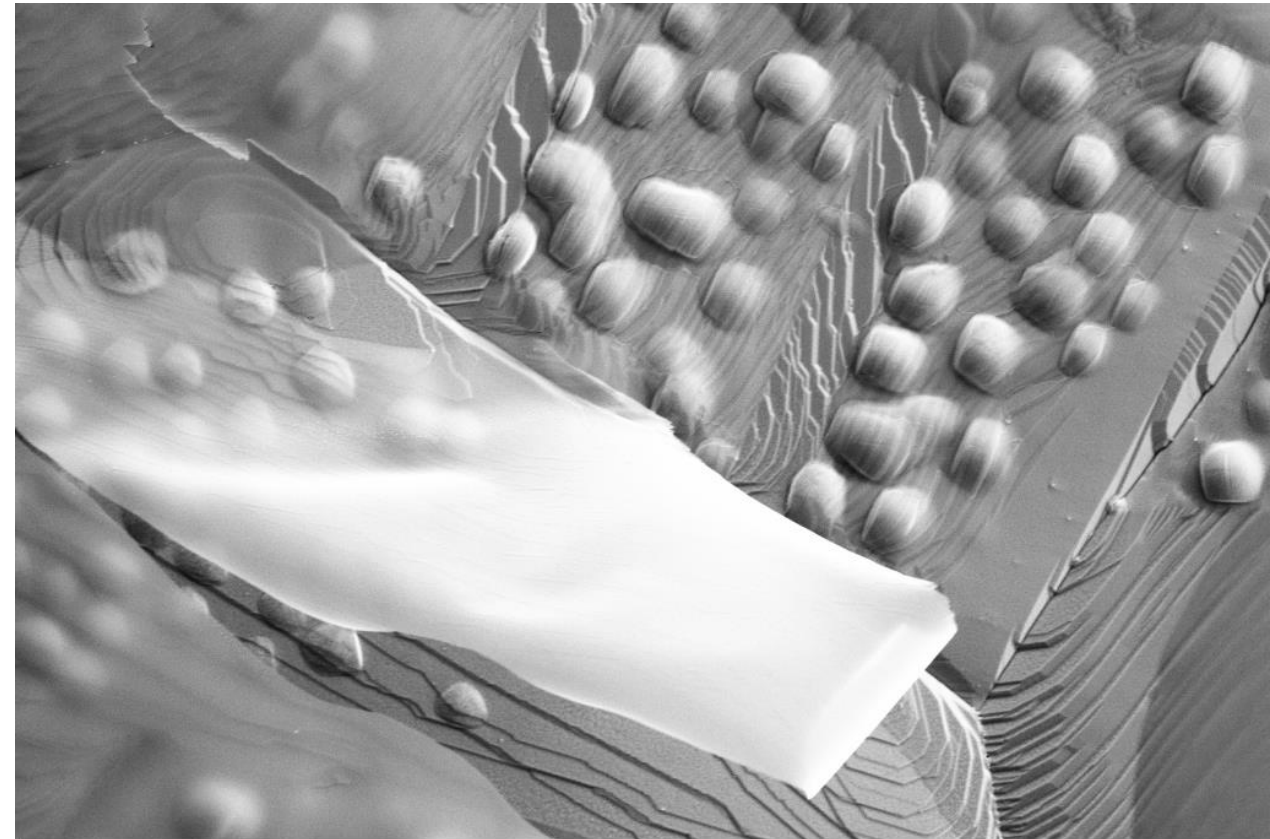
WD = 8.2 mm

Date: 31 Oct 2023



Sample ID = Cu\_irradSept23\_before\_plasma\_

Mag = 3.00 K X



EHT = 5.00 kV

Signal A = SE2

Catarina Serafim

WD = 8.2 mm

Date: 31 Oct 2023

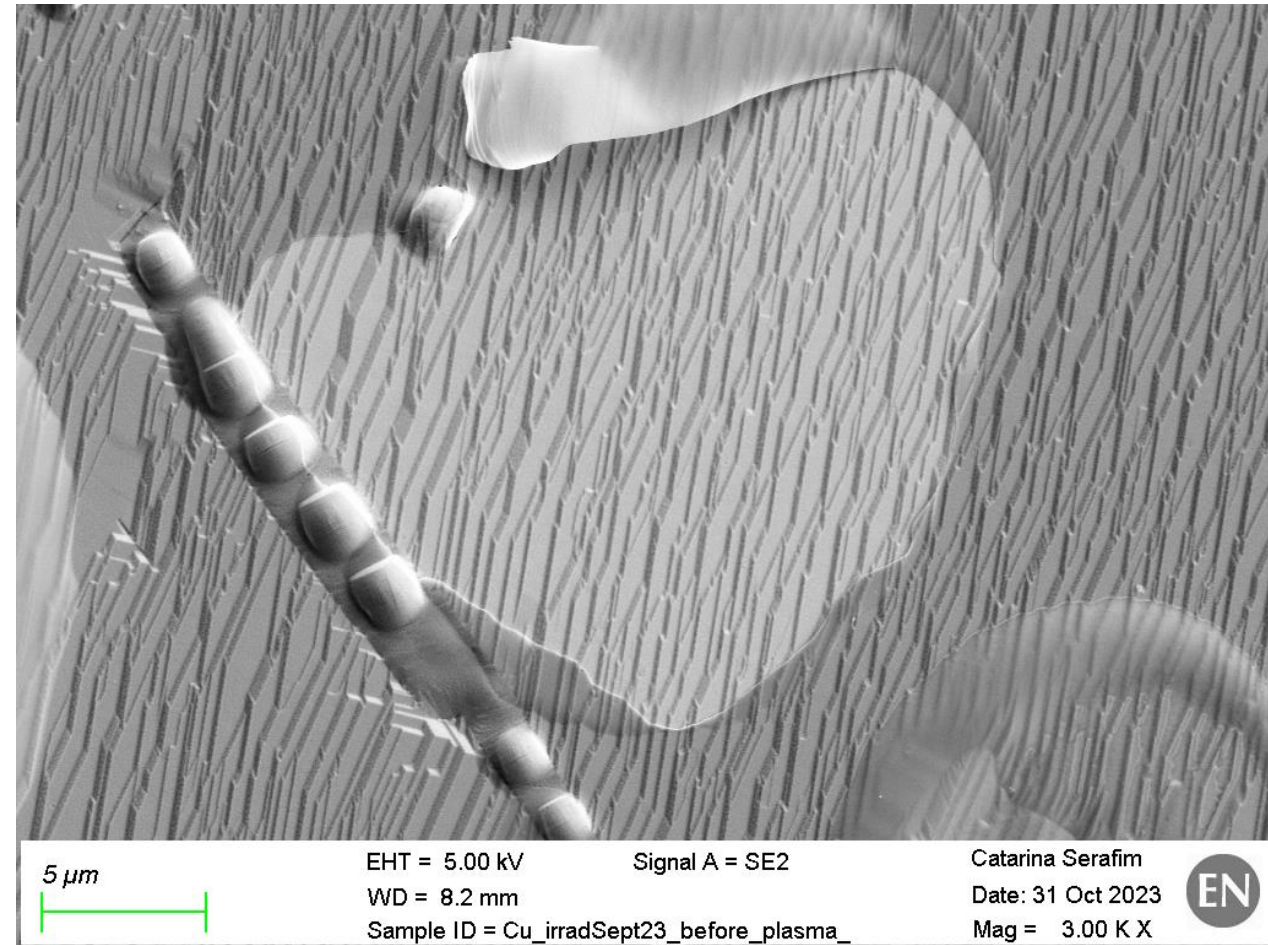
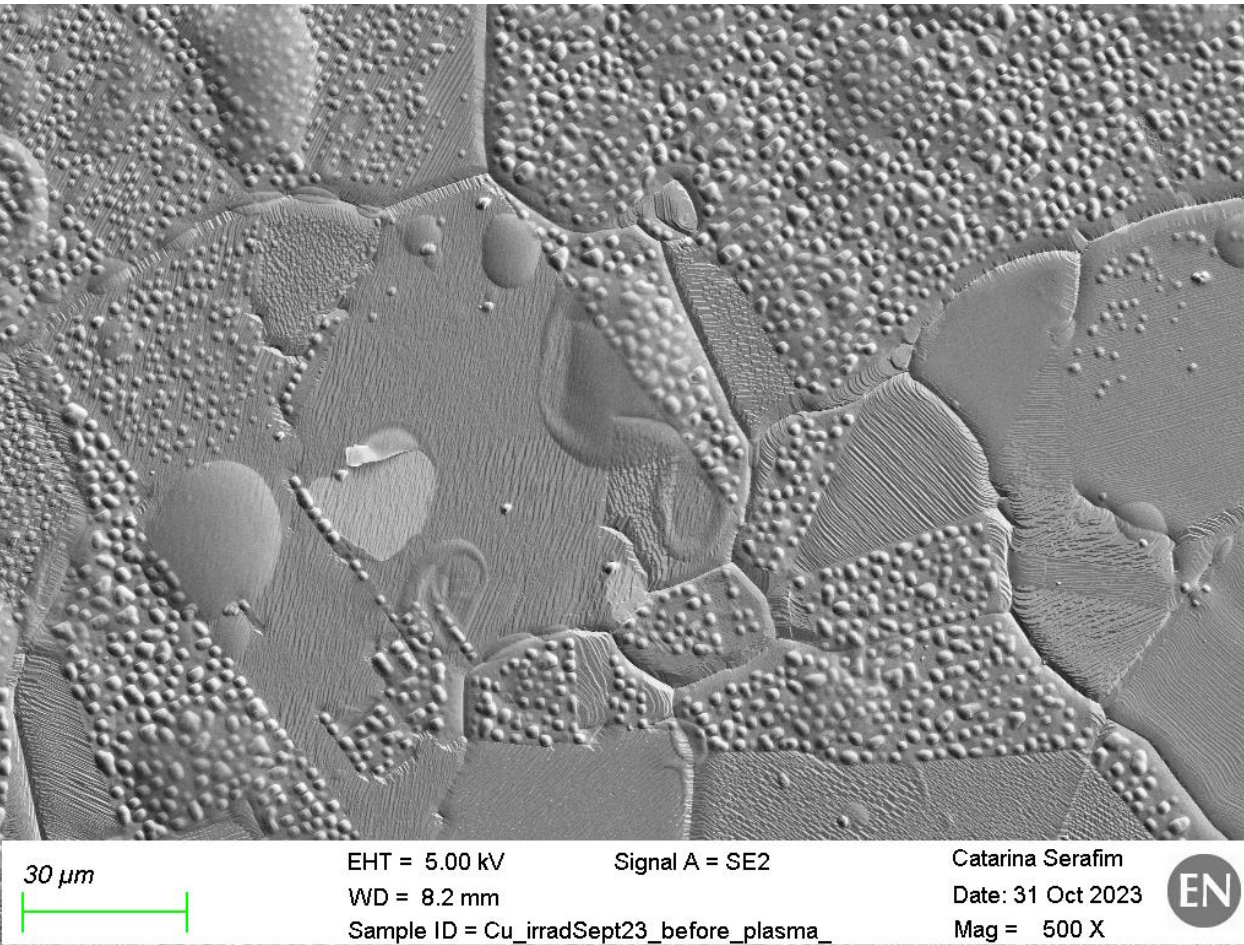


Sample ID = Cu\_irradSept23\_before\_plasma\_

Mag = 3.00 K X

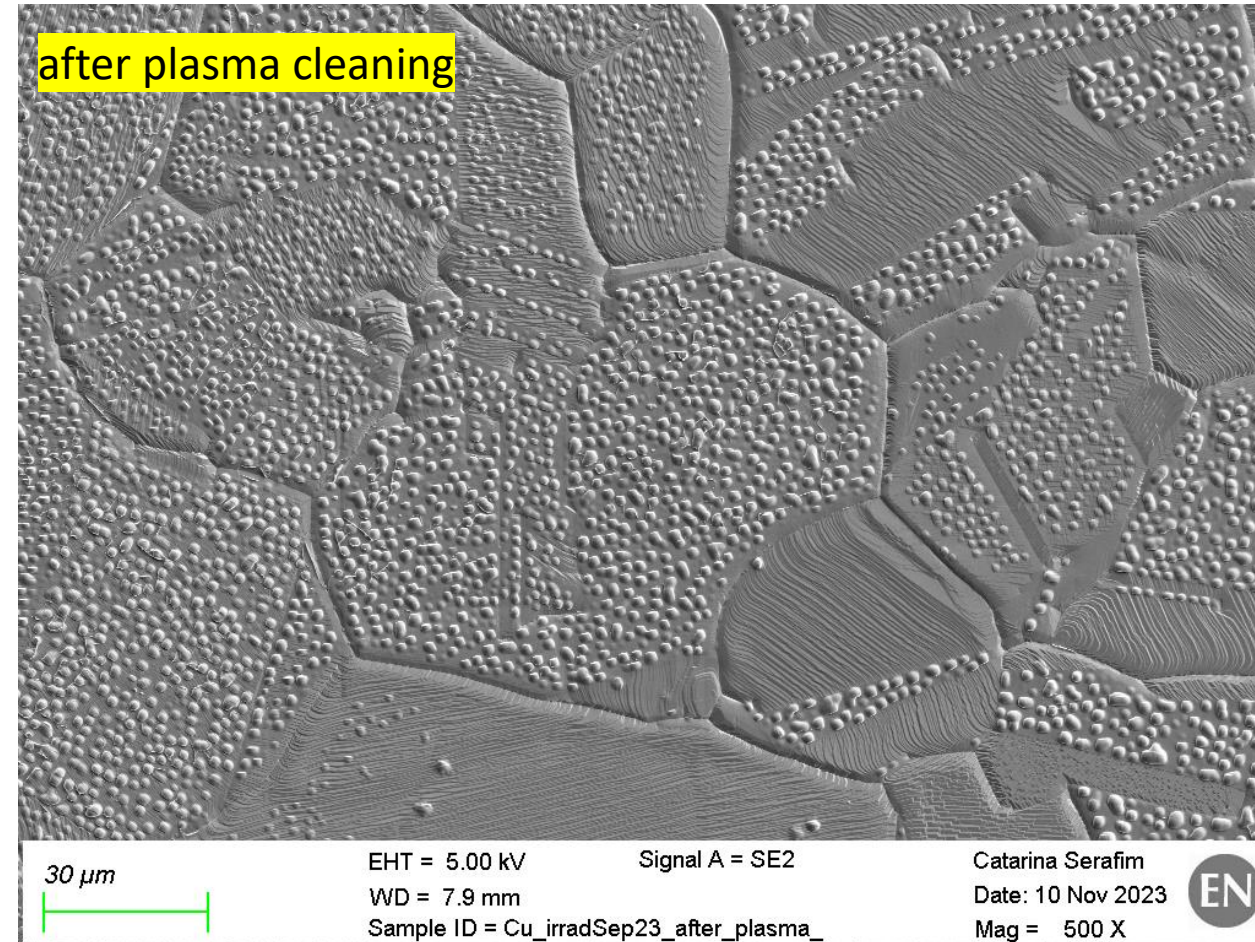
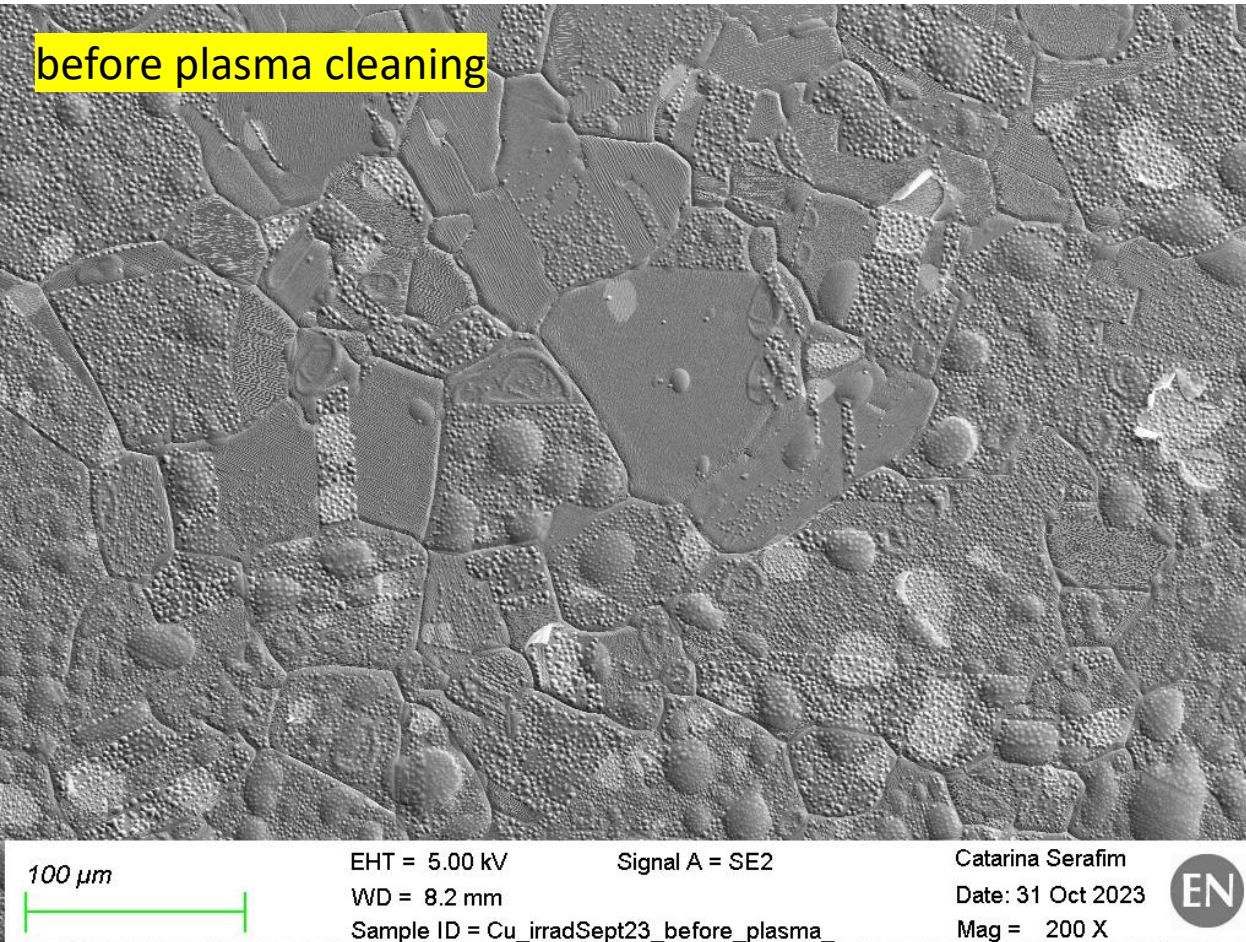


SEM Images (secondary electron detector) of Cu-OFE electrode after being exposed with H- beam before plasma cleaning



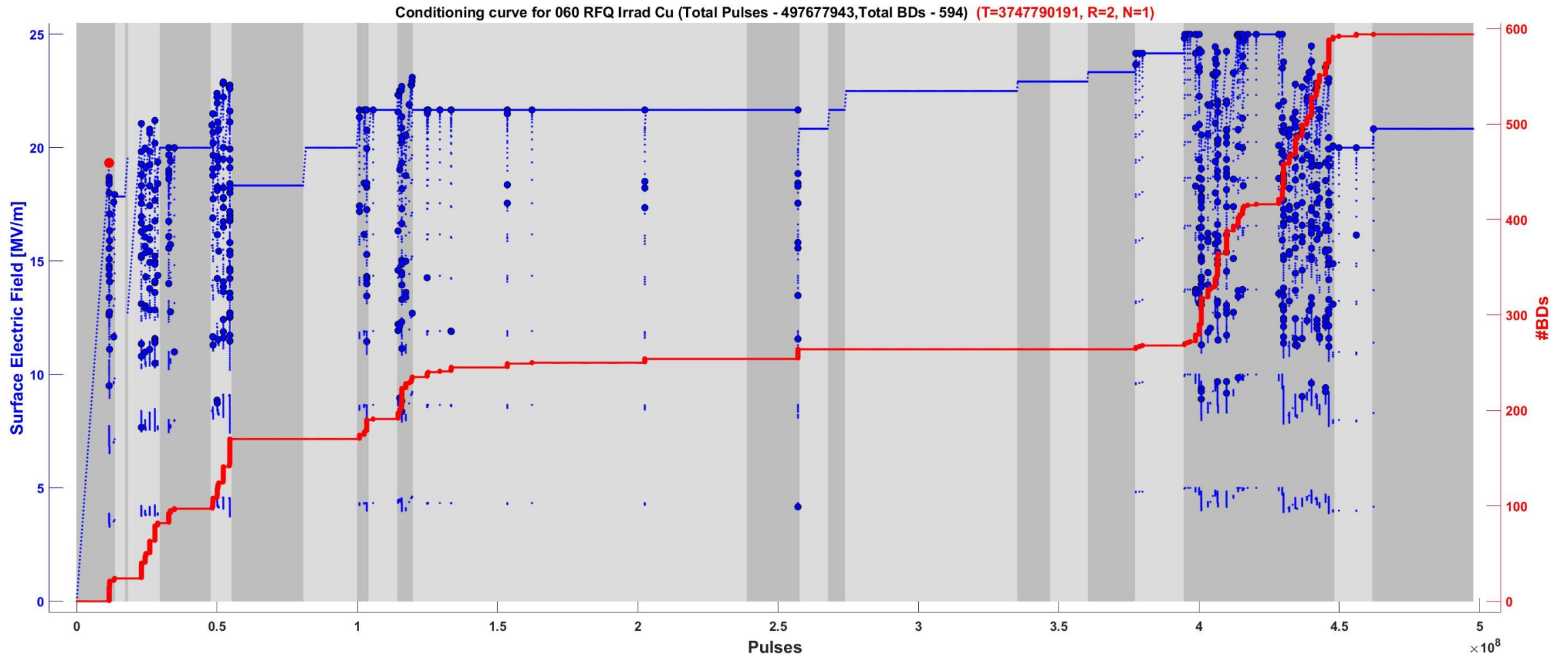


SEM Images (secondary electron detector) of Cu-OFE electrode  
after being exposed with H- beam



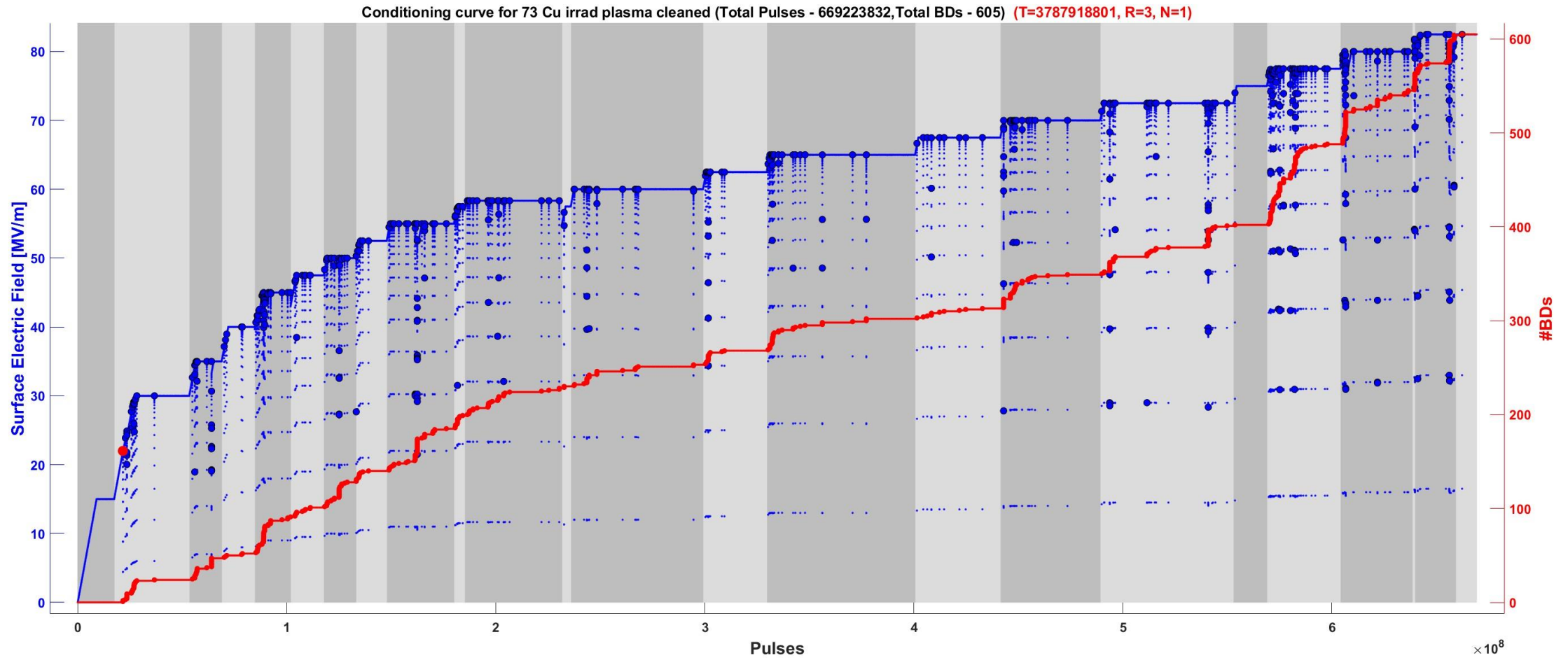


# Cu-OFE electrodes after irradiation (not plasma treated)





# Cu-OFE electrodes after irradiation and plasma cleaning

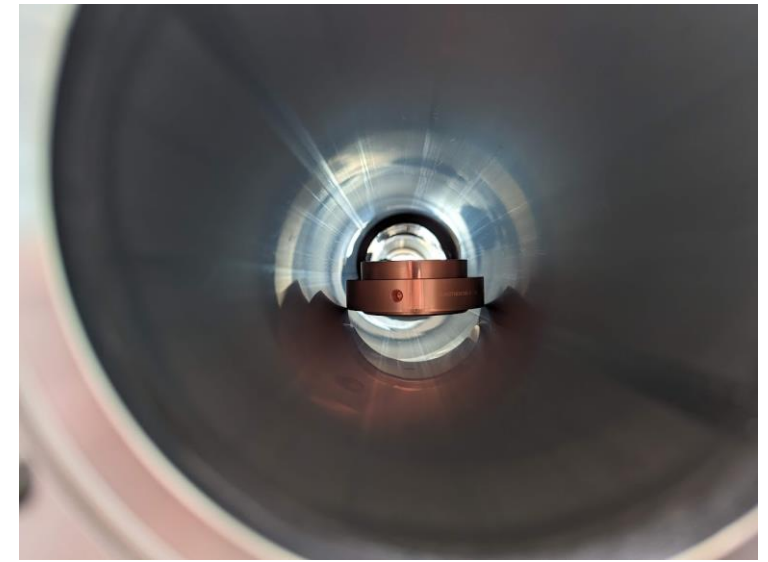




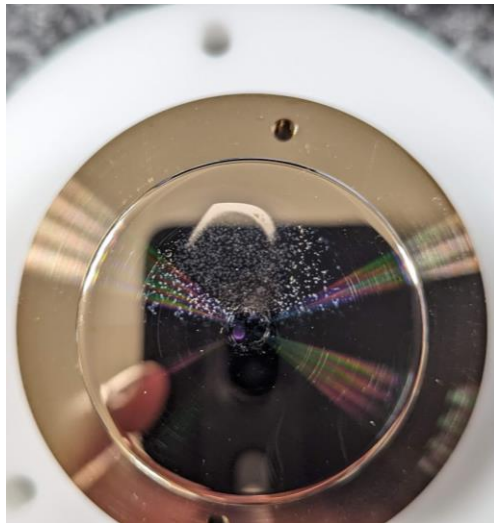
In general, some fragments of the C layer are still visible in the surface. Chemical analysis was performed in different areas before and after plasma cleaning.

- Before plasma treatment we had 9-12% of C
- After plasma cleaning we have 1-3% of C

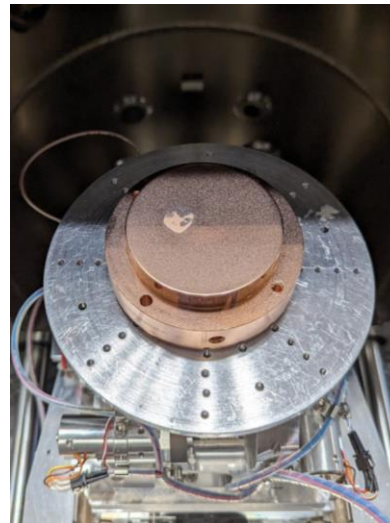
Cu-OFE and CuBe2 have shown good conditioning results after the treatment. Next Step will be to test SS316, which achieved 120 MV/m (non-irradiated).



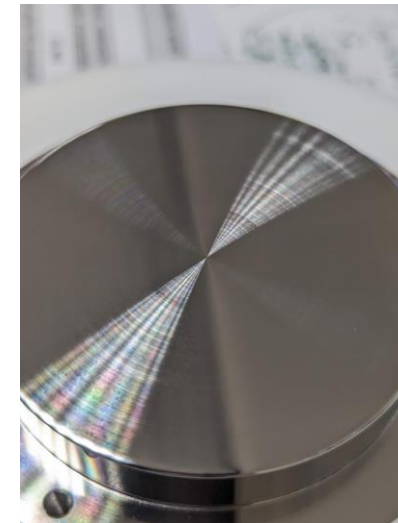
### Plasma treated electrodes 2023/2024



*CuBe2*



*Cu-OFE*



*SS316*



**Thank you**