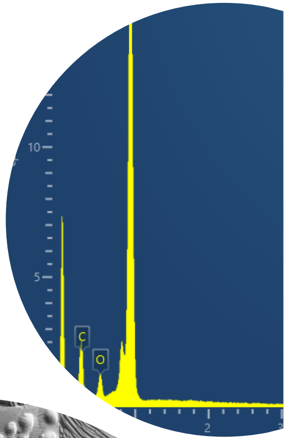
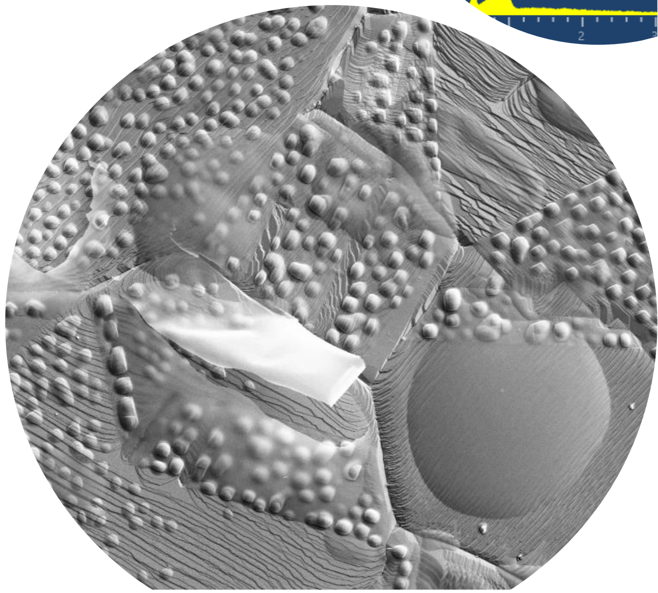
CLIC Mini Workshop

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



12th December, 2023
Switzerland

UNIVERSITY OF HELSINKI

Motivation/Selection of materials

➢ **Finding a new material with a better performance to a future manufacturing of a new RFQ.**

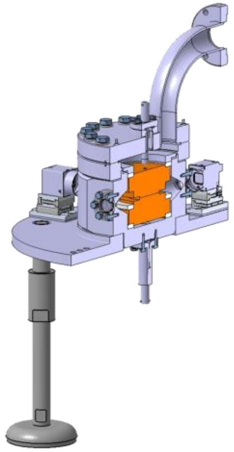
- Need to understand origin of enhanced breakdown rate, and find a mitigation
- Can breakdowns be correlated with beam losses ?
- Can blistering influence the lower performance?

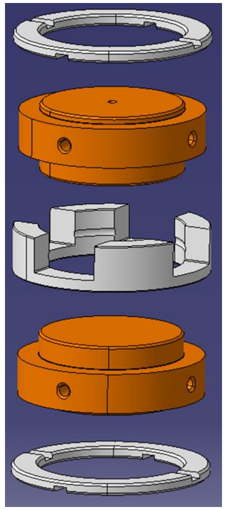
Selected Materials	Source of manufacturing	Quantity
Cu-OFE	Purchase from external company	2 pairs of anode-cathode electrodes
TiAl6V4	Machined at CERN Workshop	
Nb	Machined at CERN Workshop	
CuCrZr	Machined at CERN Workshop	
CuBe2	Purchase from external company	
Ta	Machined at CERN Workshop	
SS316	Machined at CERN Workshop	

Testing
- 1 - Irradiation with H- beam
- 2 - High Voltage Pulsing testing with LES system

Materials were selected based on their:

➢ Usability for meter-long high gradient RF cavities
➢ Potential resistance to blistering
➢ Resistance to breakdown phenomena

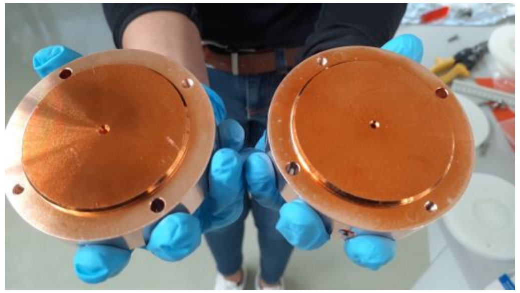




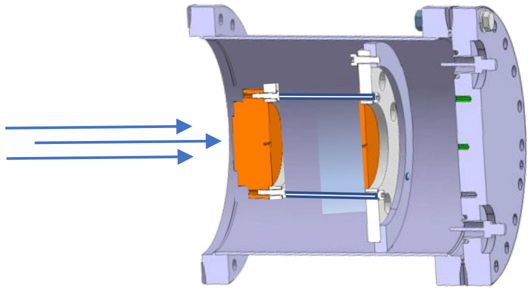
Procedure

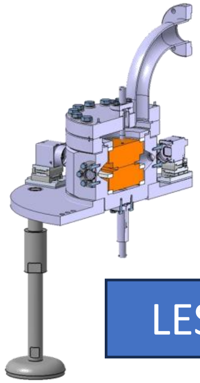
1st pair of electrodes

Treatment of electrodes



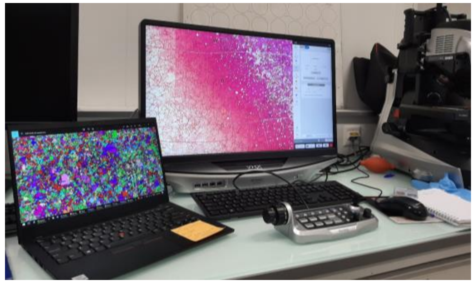
H- irradiation





LES tests

Analysis

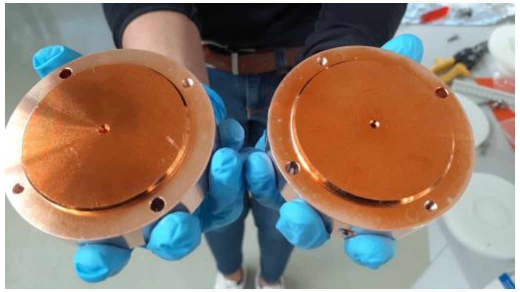


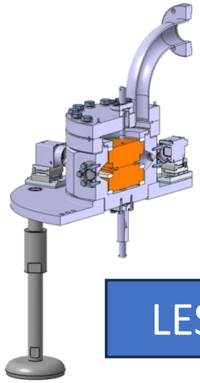
Before testing electrodes have been through a preparation phase:

- Manufacturing/Machining
- Metrology and SEM finishing surface analyses
- Surface cleaning
- Thermal treatments

2nd pair of electrodes

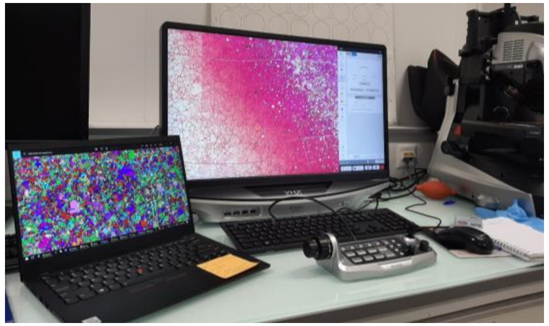
Treatment of electrodes



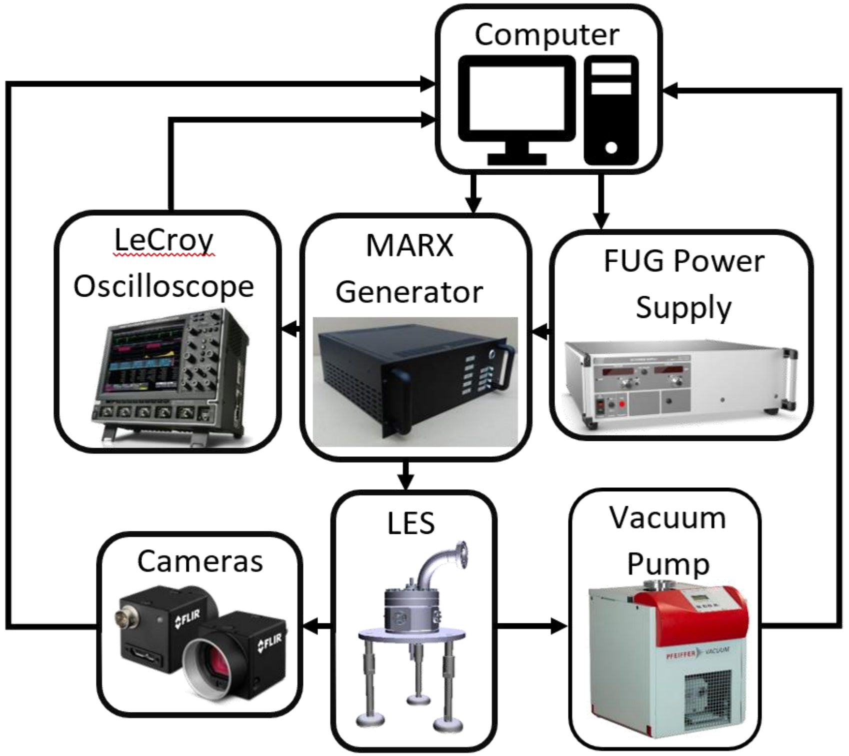


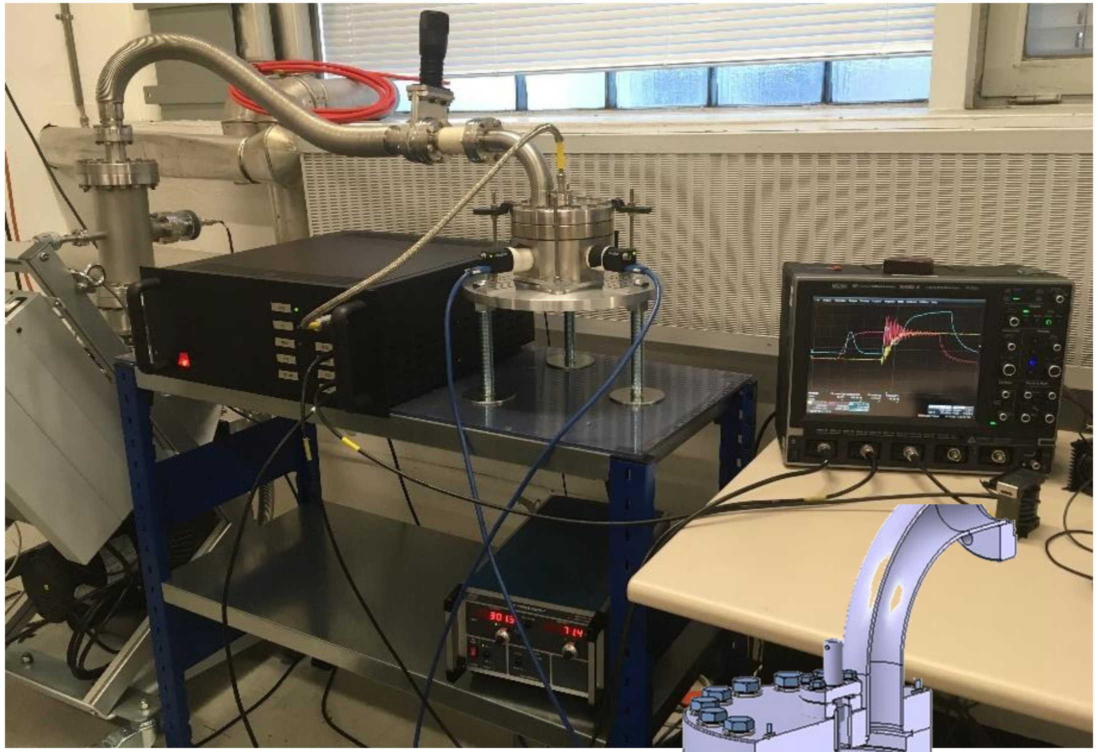
LES tests

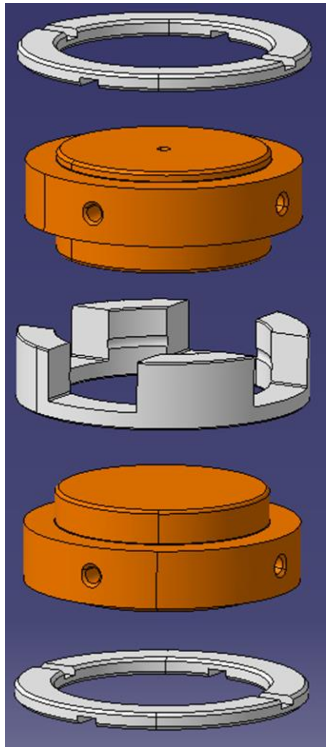
Analysis

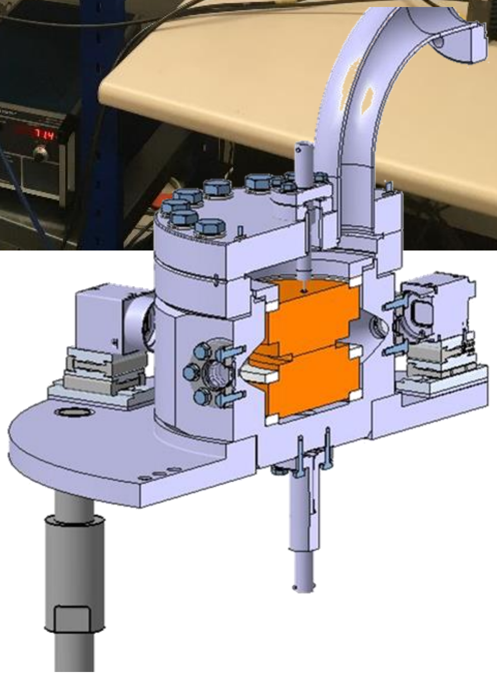


Pulsed DC Large Electrode System Setup



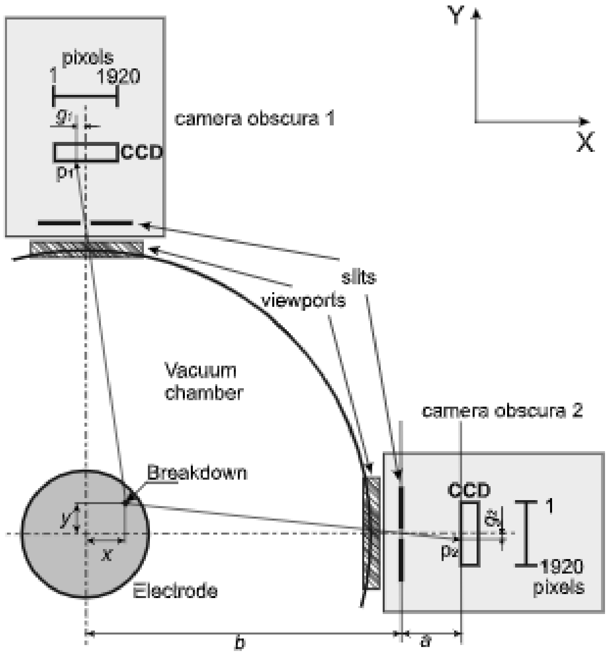




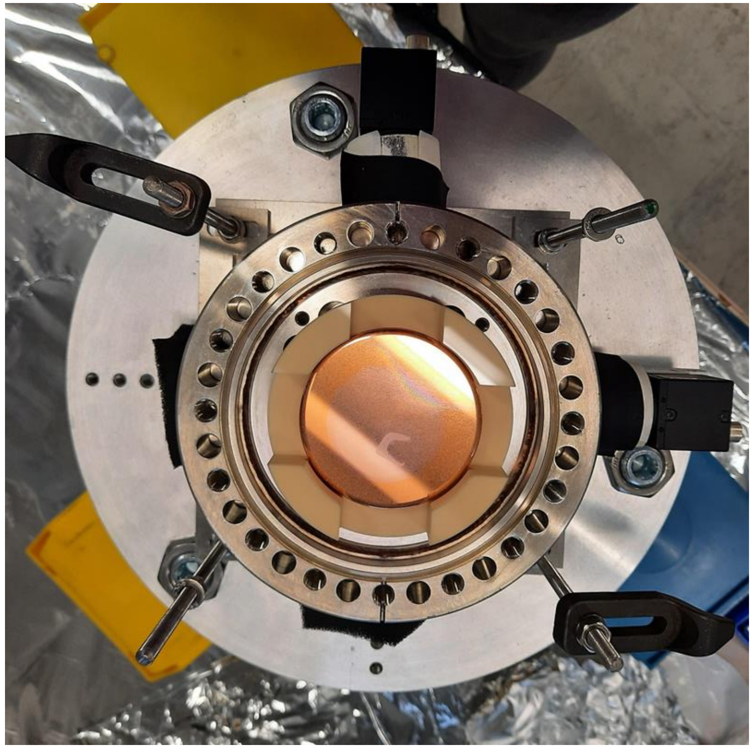


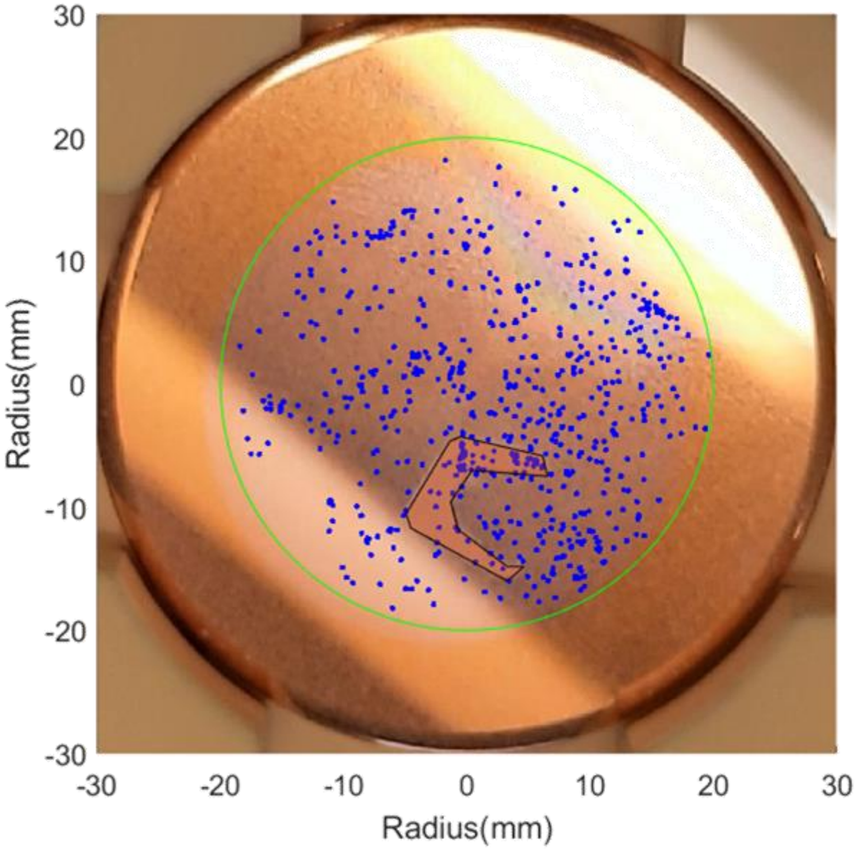
- The MARX generator can pulse up to a rep rate of 6kHz.
- Measurements of the voltage and current pulses are done.
- Breakdowns are detected both form the pulse shape and optically.

Breakdown Location

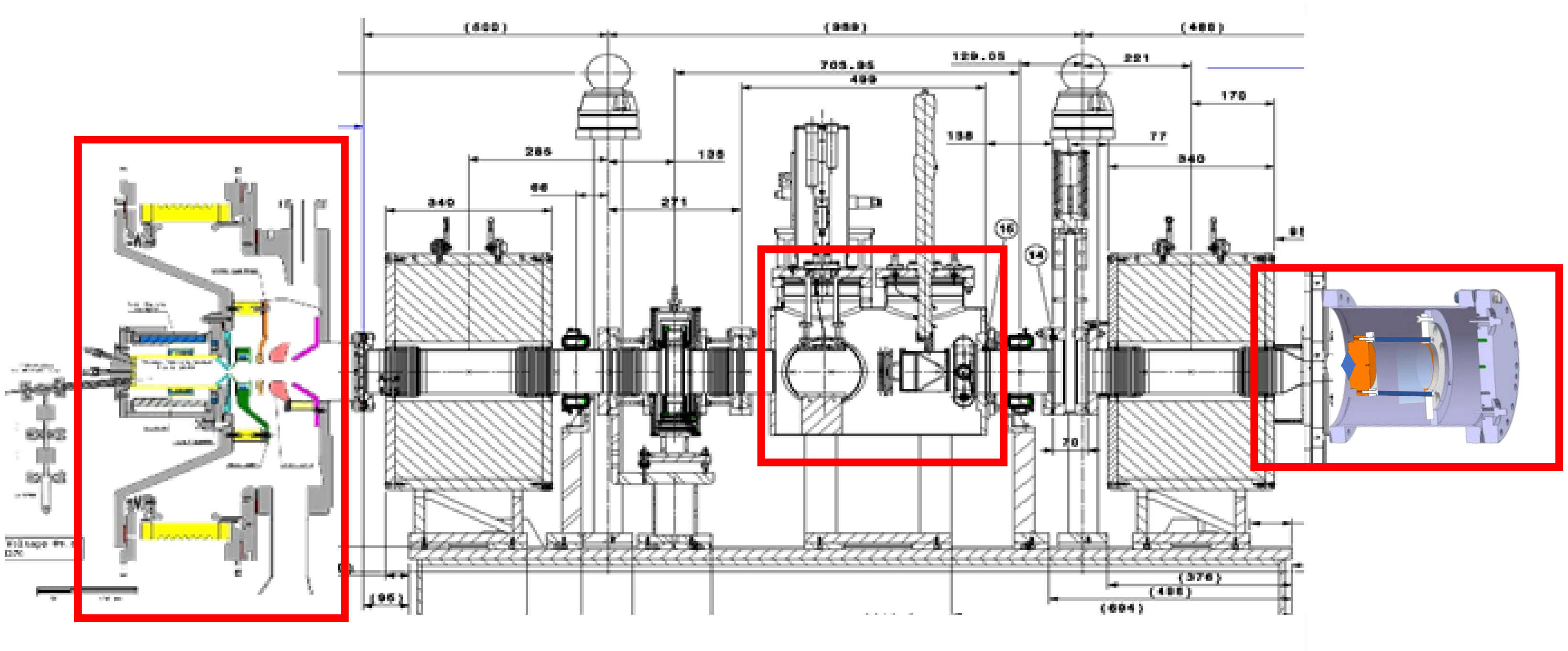


(2019). Breakdown localisation in a pulsed DC electrode system. Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment. 953. 10.1016/j.nima.2019.163079.





Irradiation setup – LINAC4 source test stand – Irradiation affects study



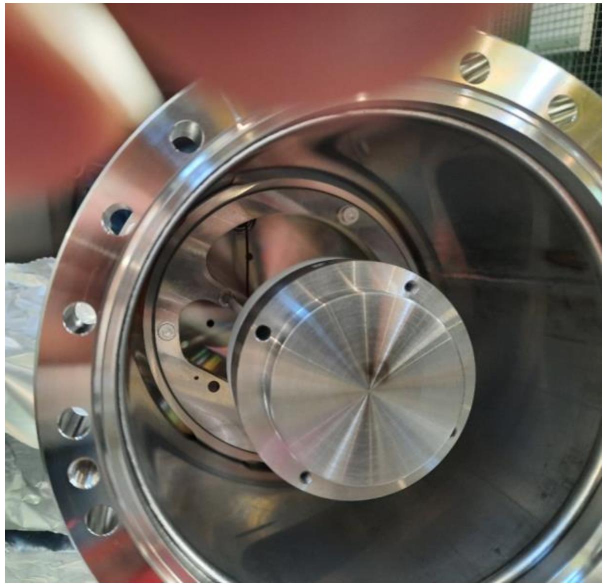
H^- Source 45 keV

Low-energy beam transport

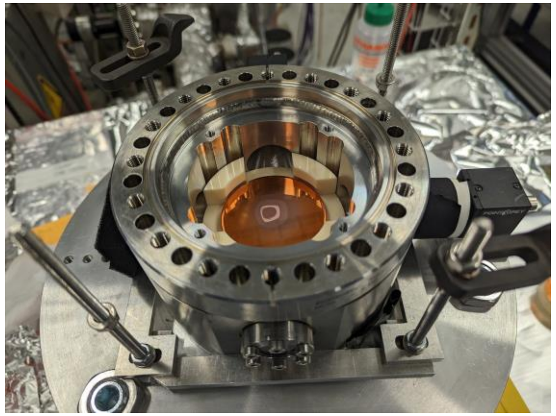
2 Steerers, 2 Solenoids

Sample holder

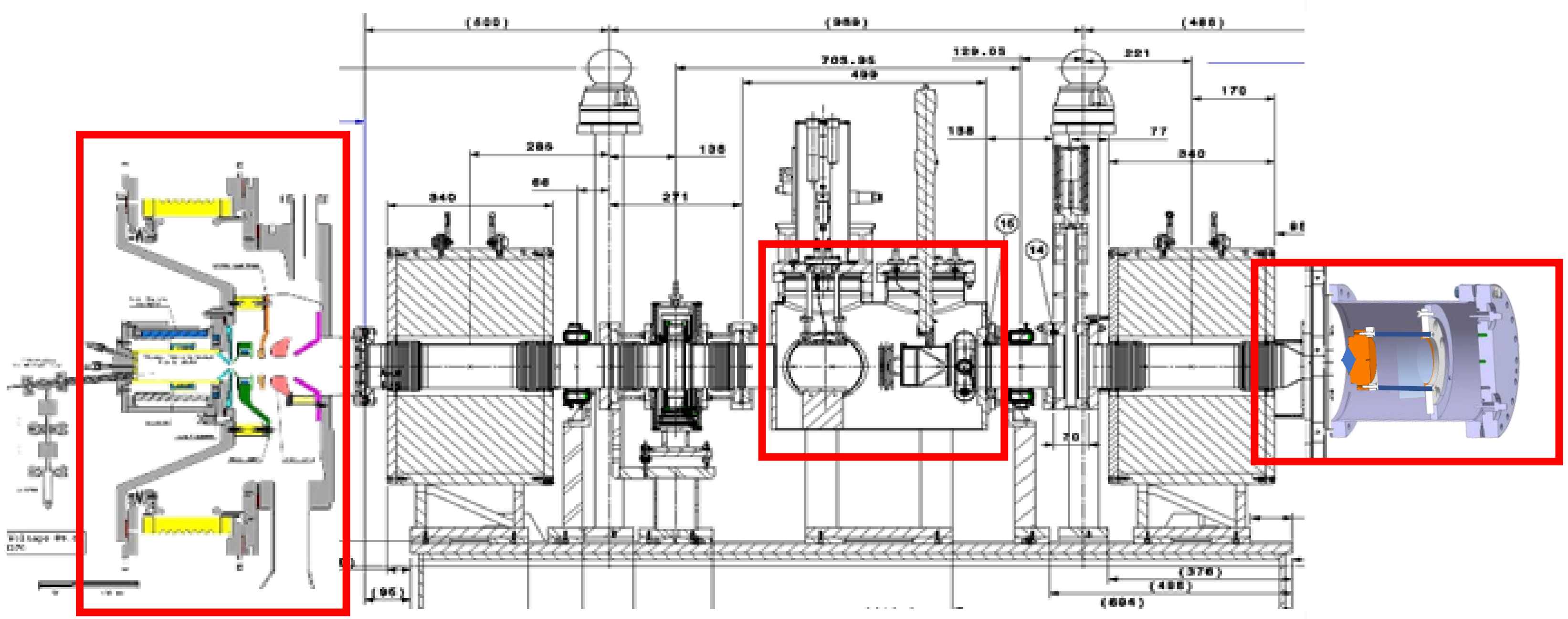
Schematic from Alessandra Lombardi



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$^-$ Source 45 keV

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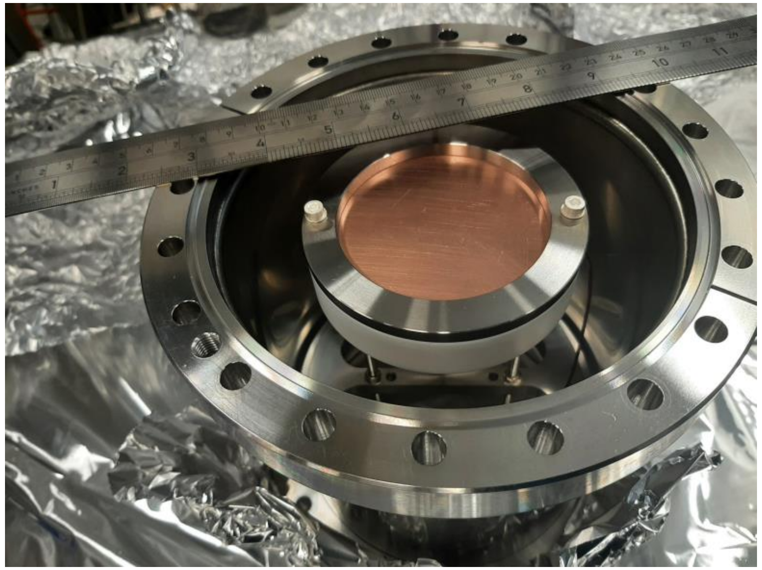
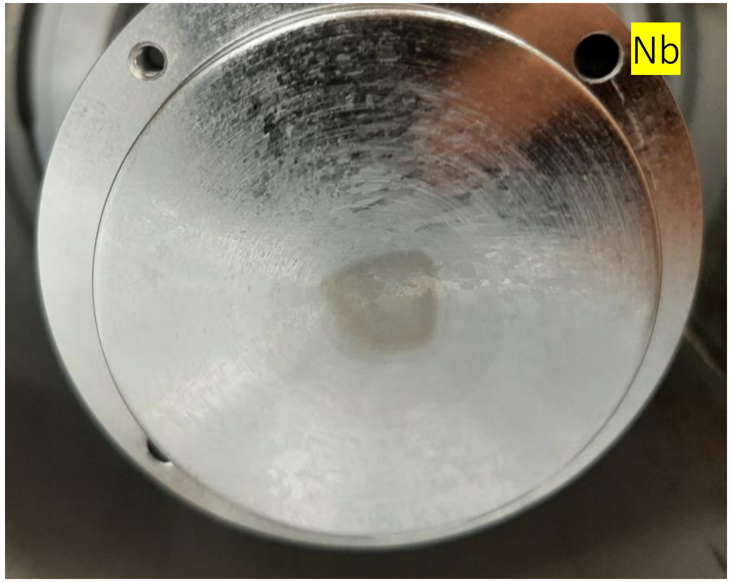
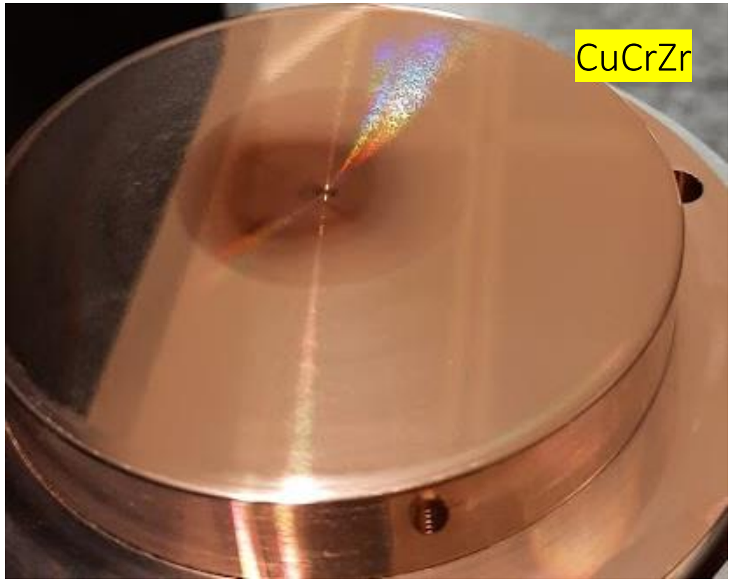
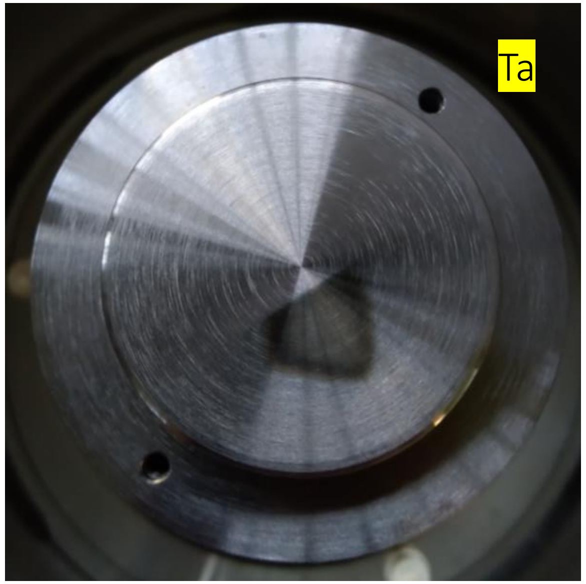


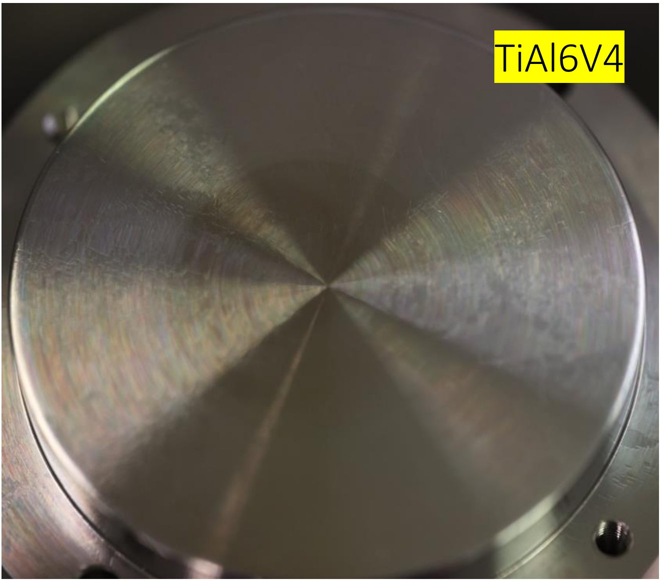
Low Energy H- beam	45 keV
Duration	40 hours
Pulse duration	600 µs
Repetition Rate	0.83 Hz
Peak current	20 mA
Deposition of particles on the target	1.2x 10^19 H$^-$p/cm^2

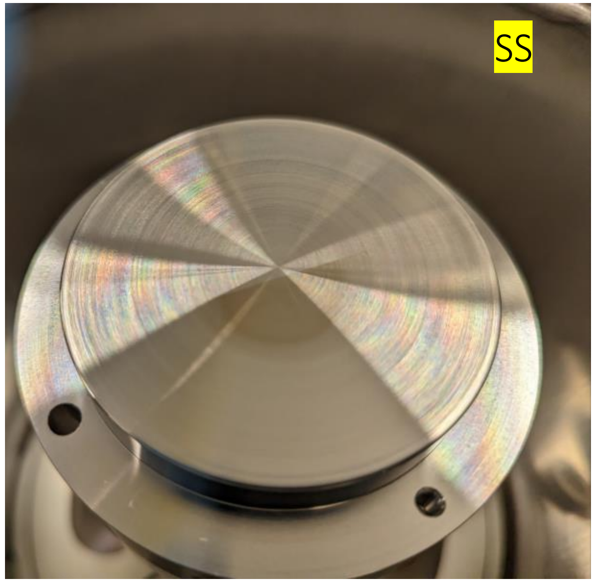
Pictures of each electrode from each material after irradiation testing.

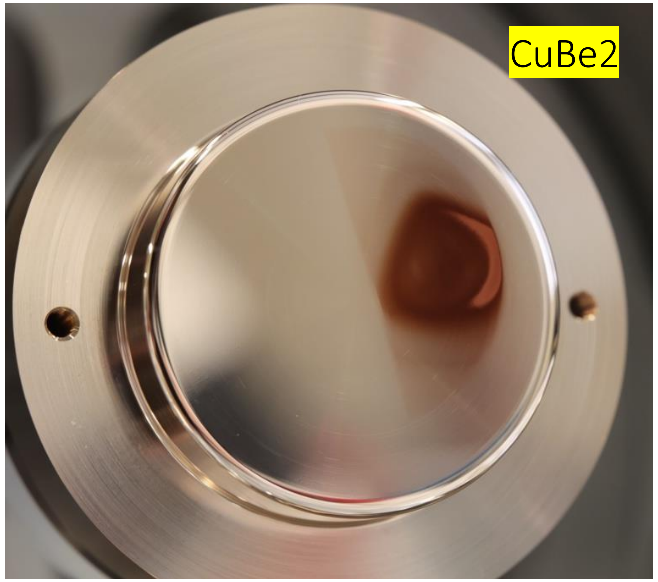


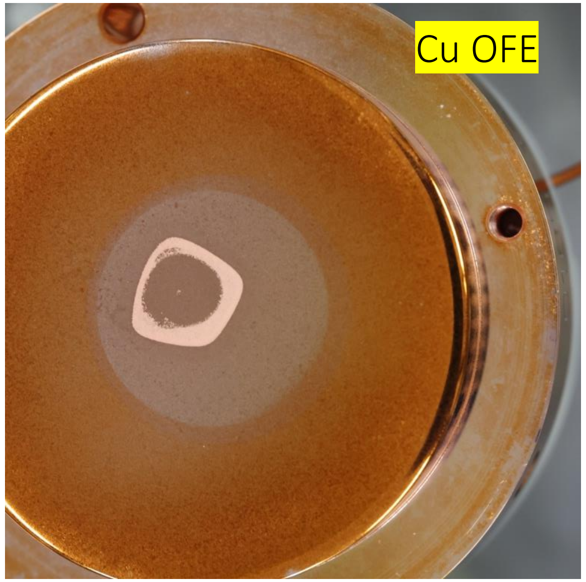




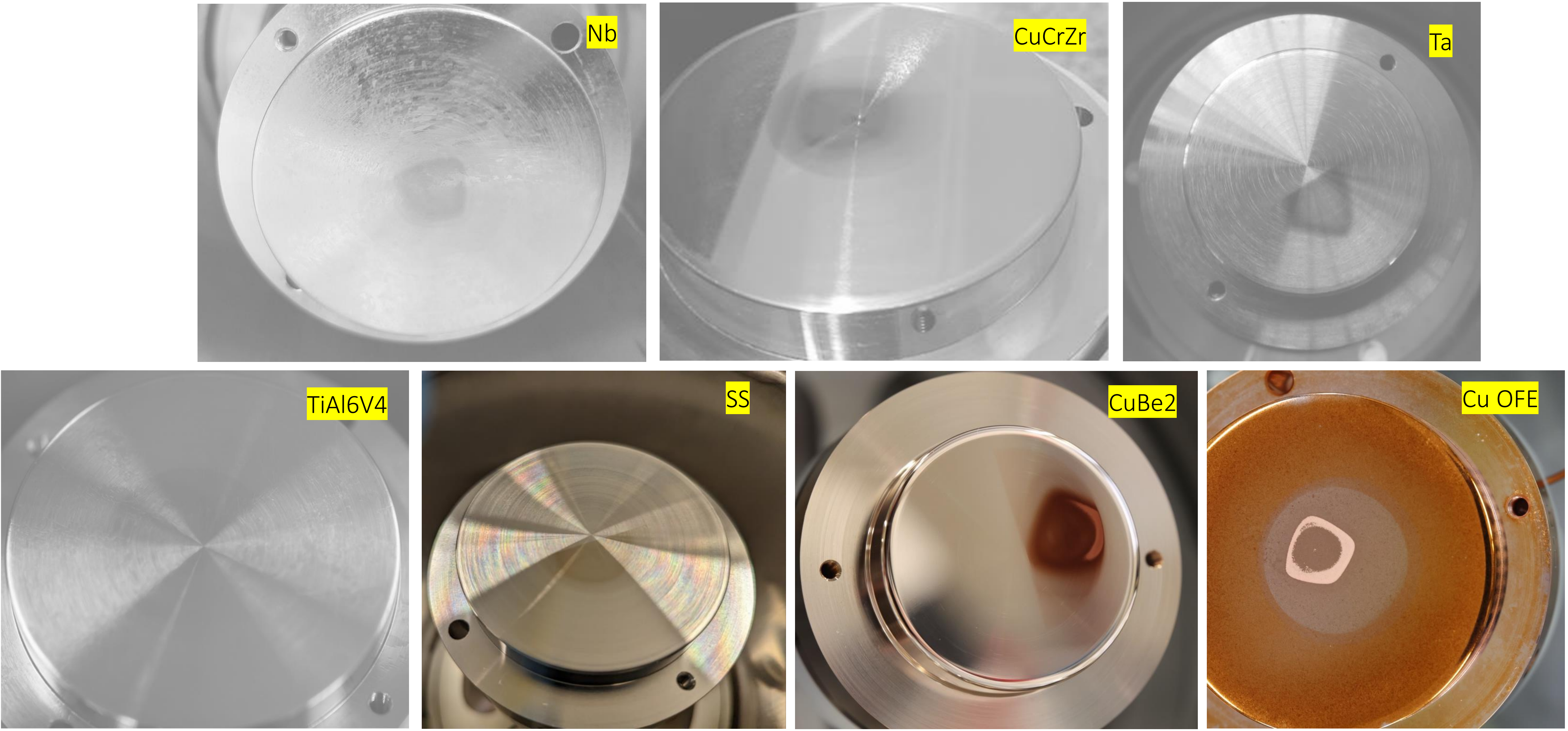






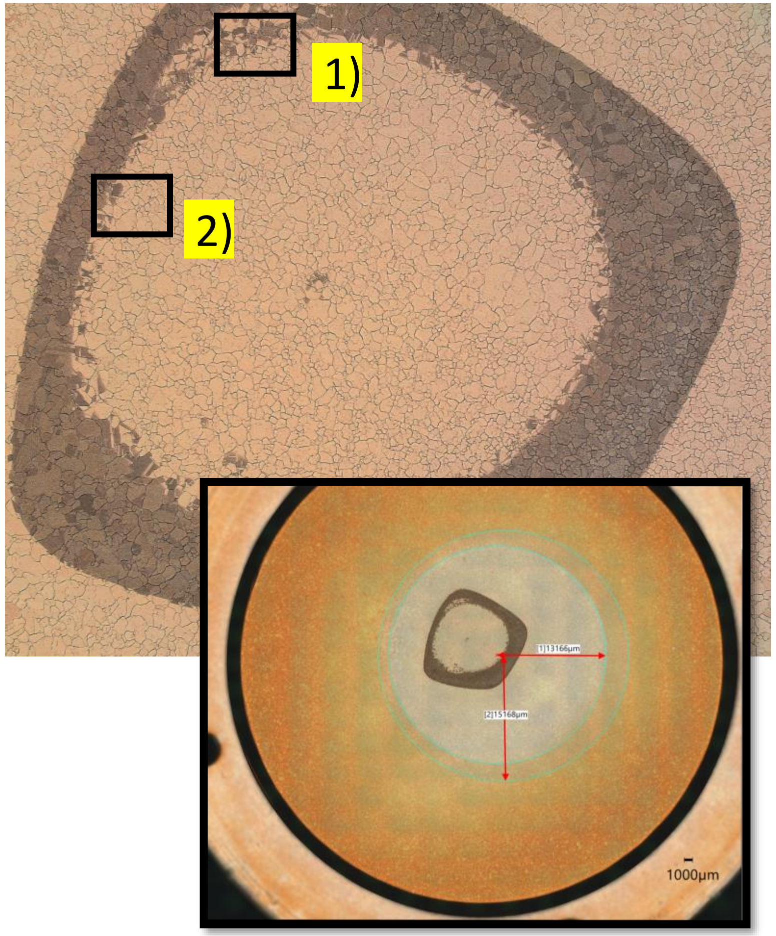


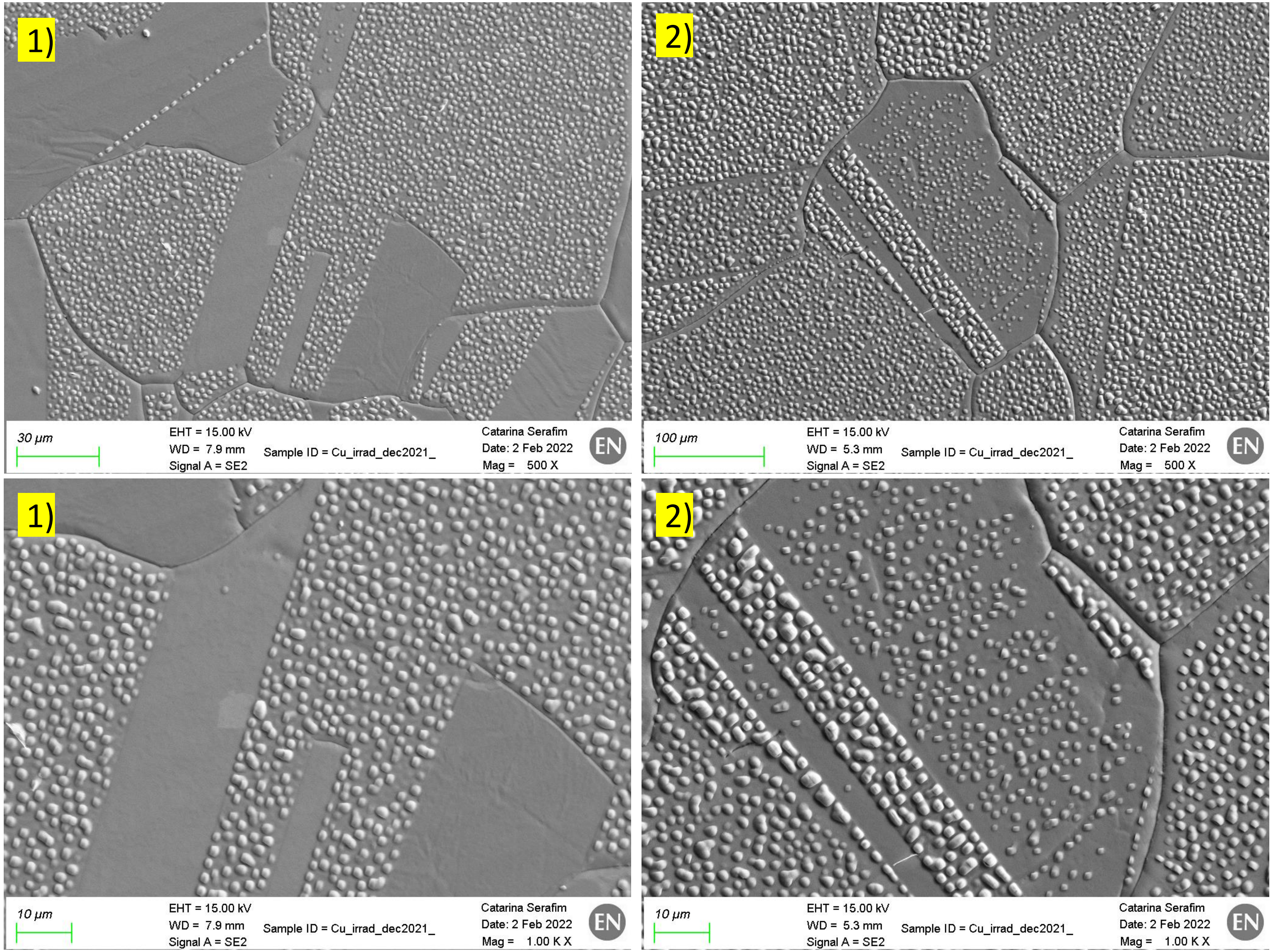
Pictures of each electrode from each material after irradiation testing.



Irradiated Cu cathode

SEM observations in different areas surrounding the irradiation zone





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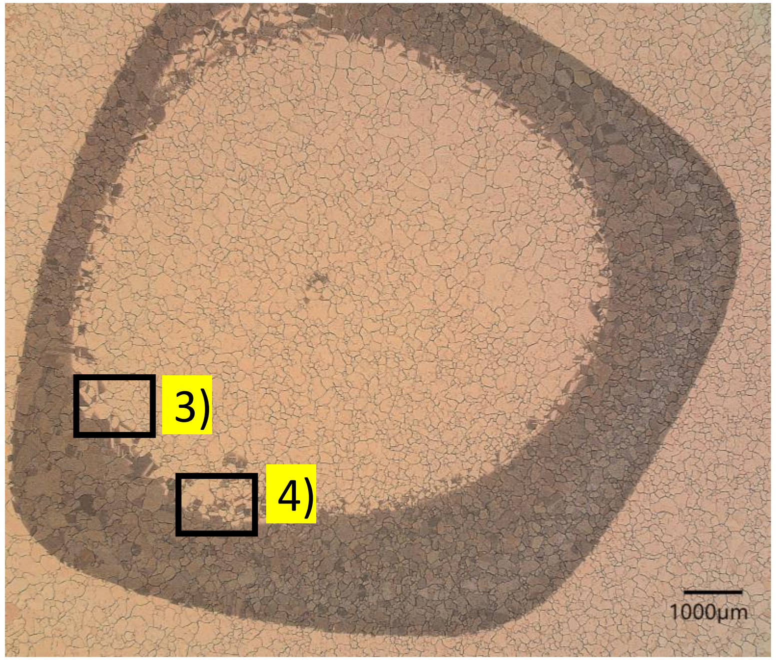
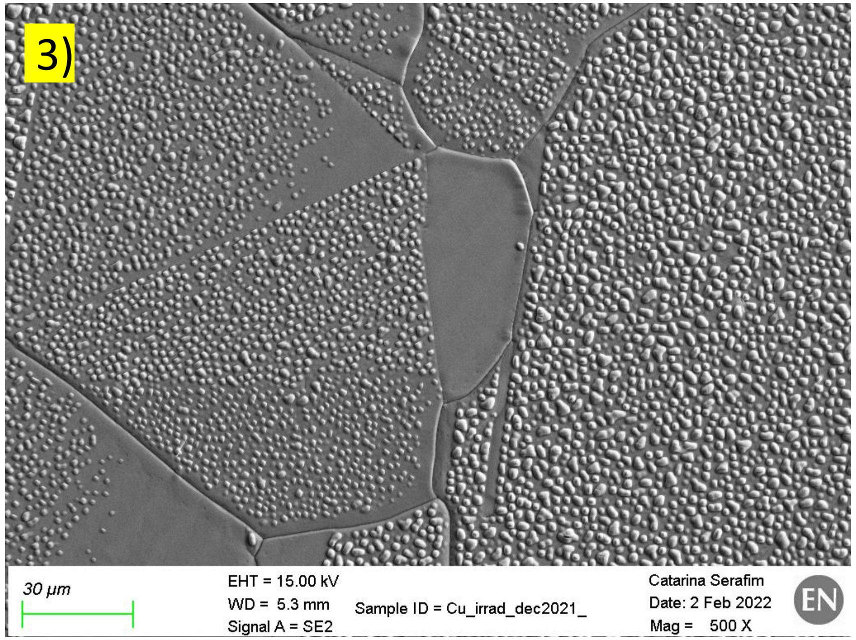
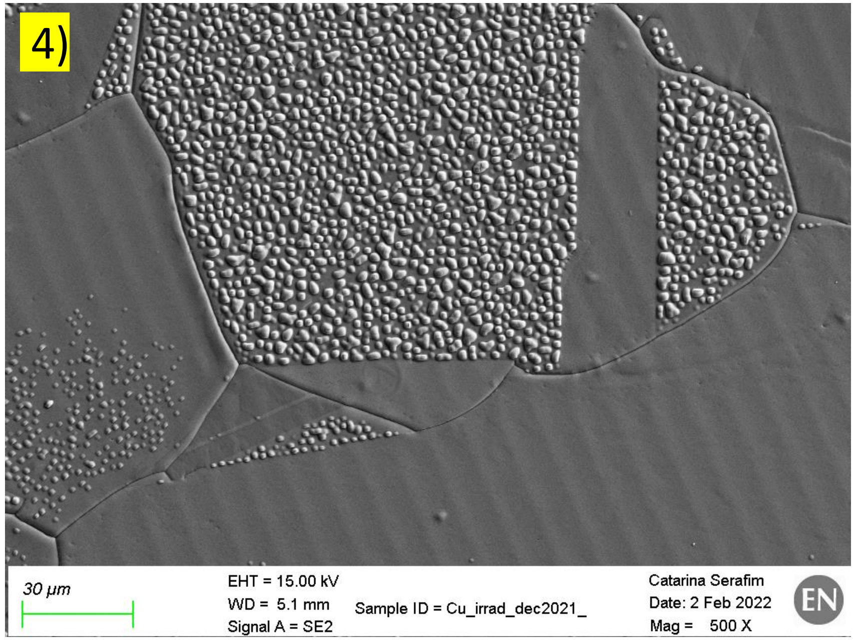
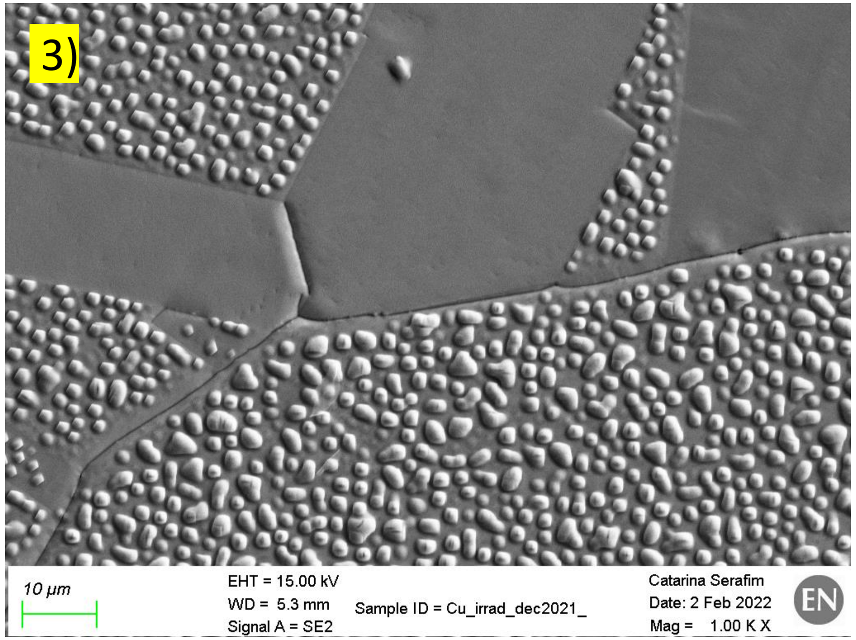
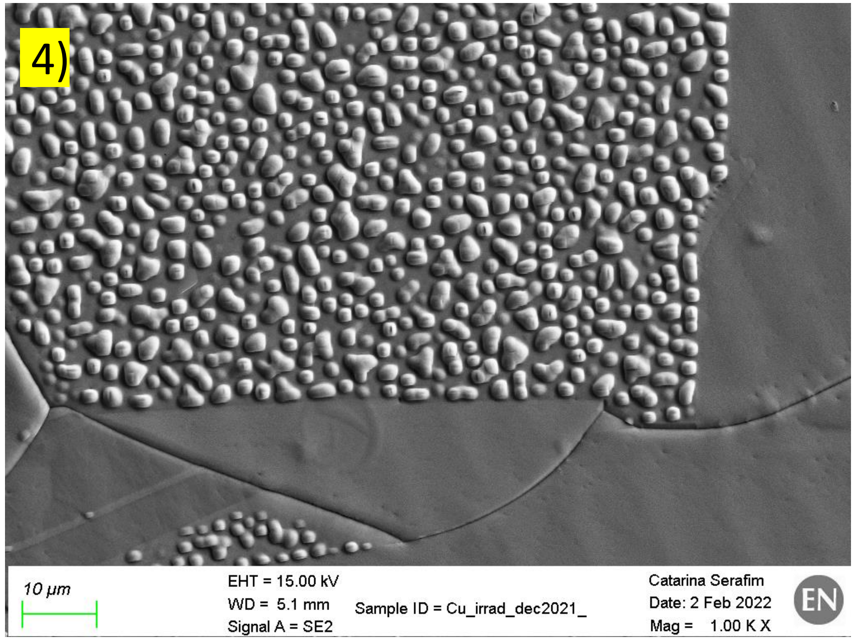


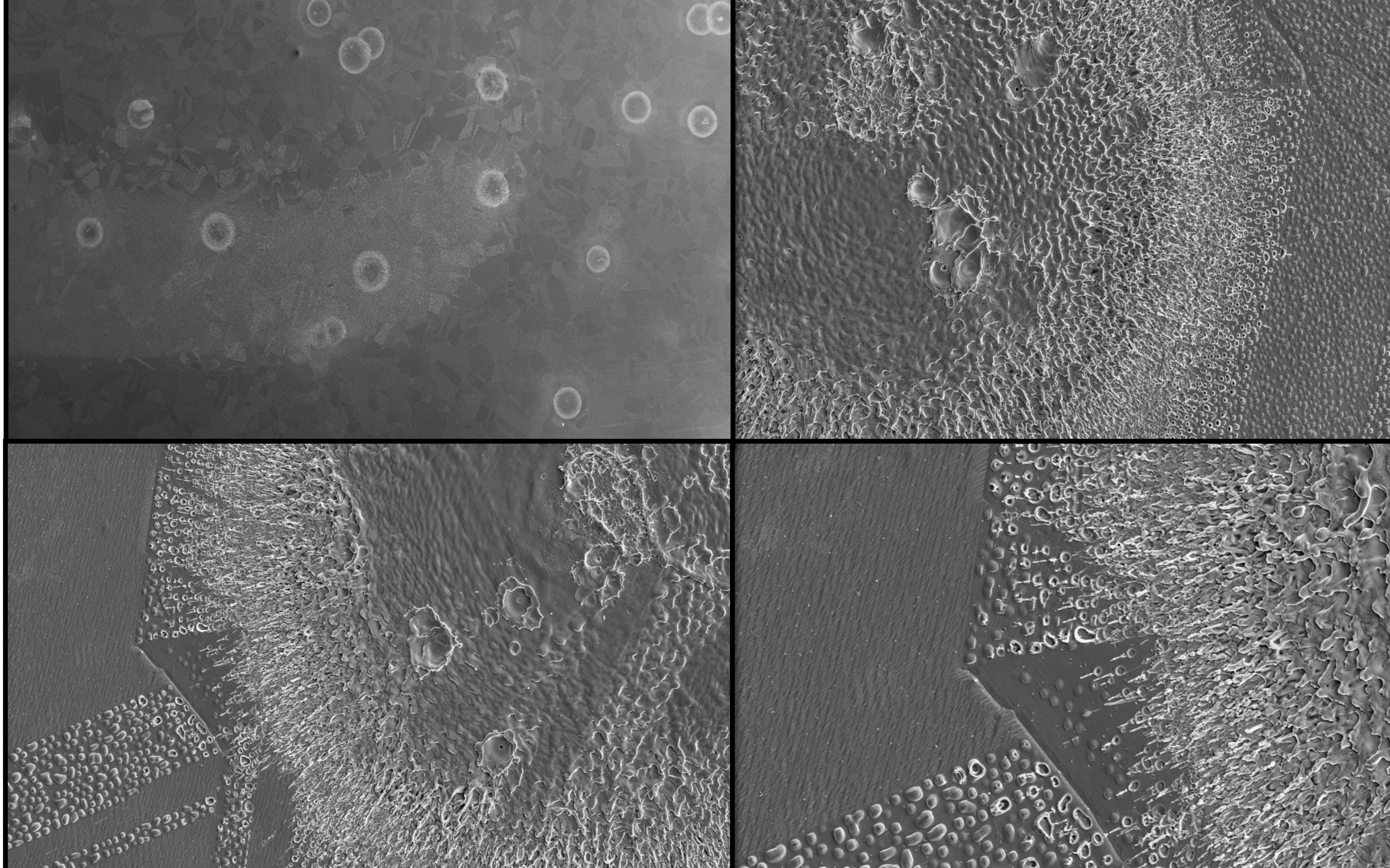




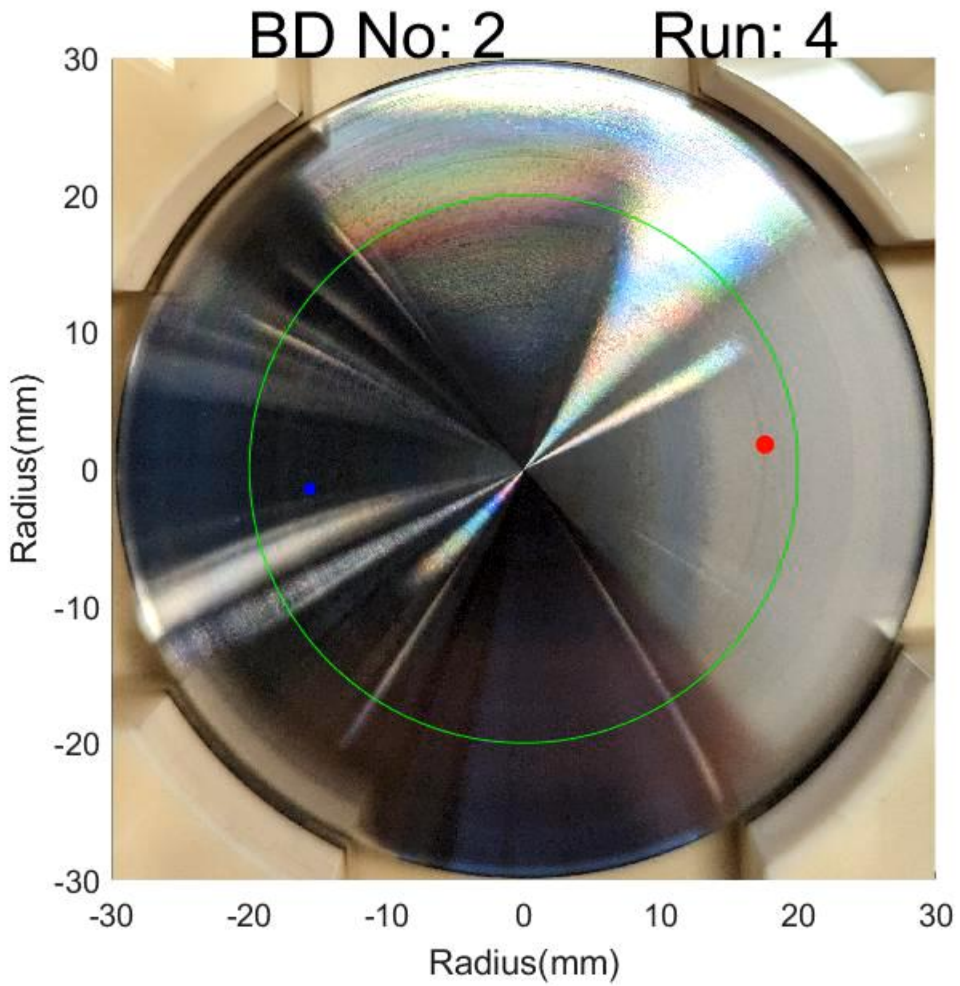


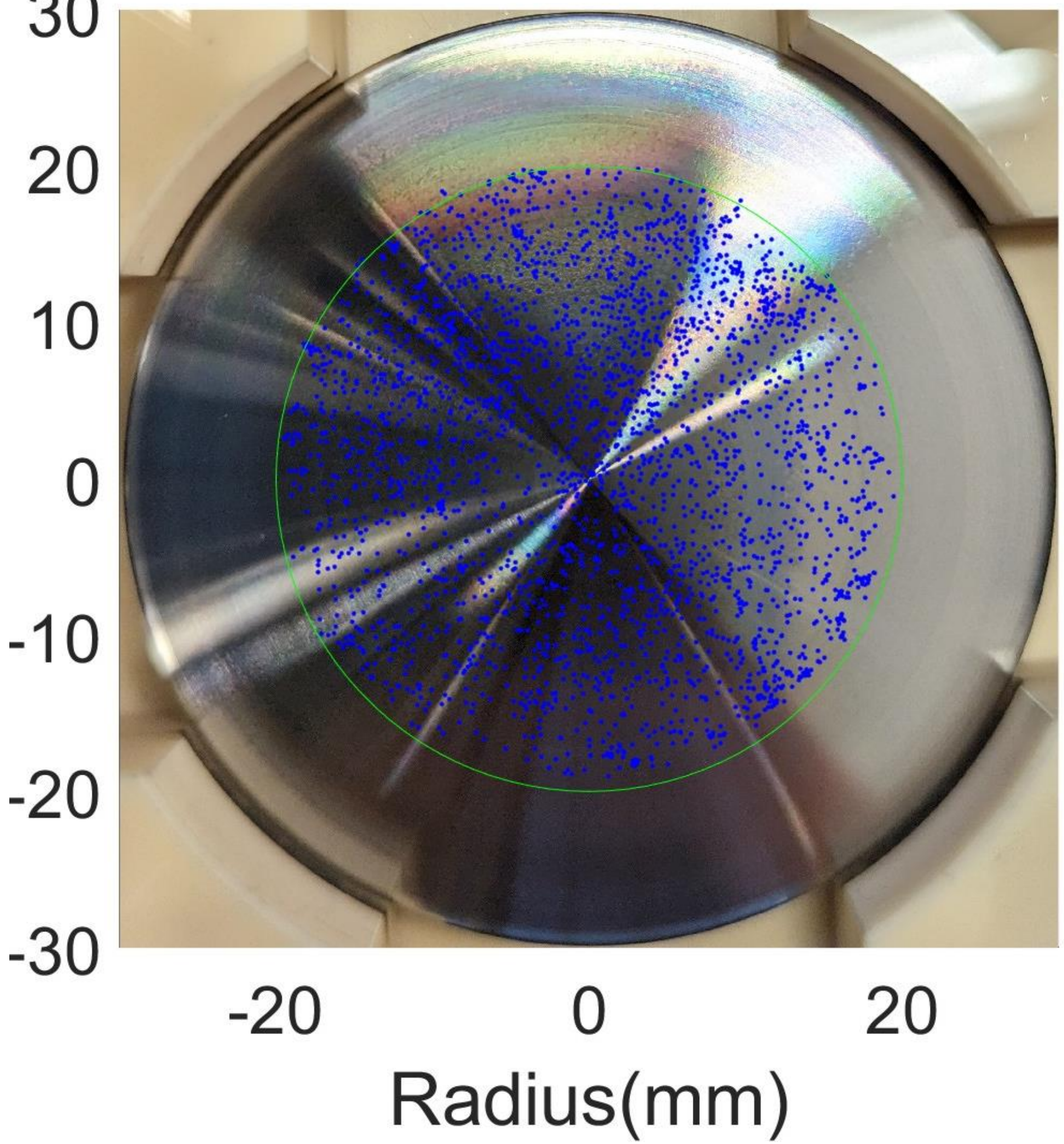


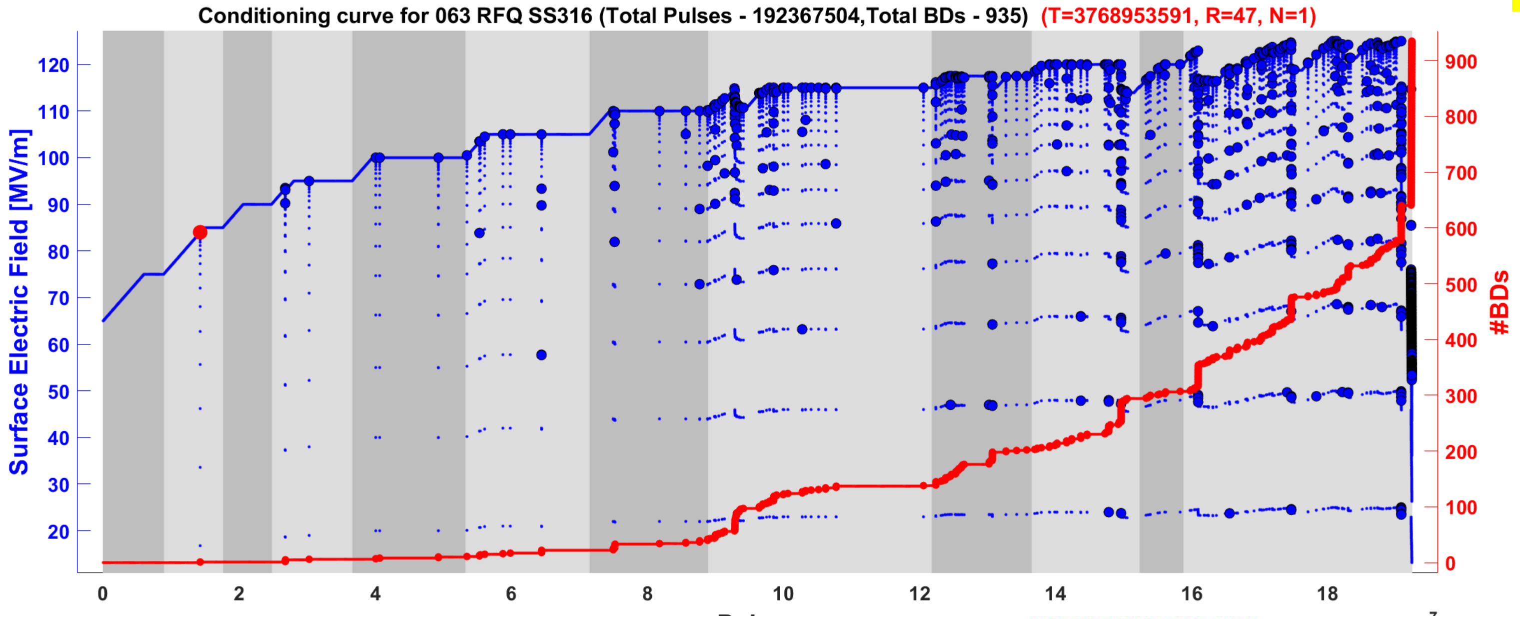
Irradiated Cu cathode, 2021

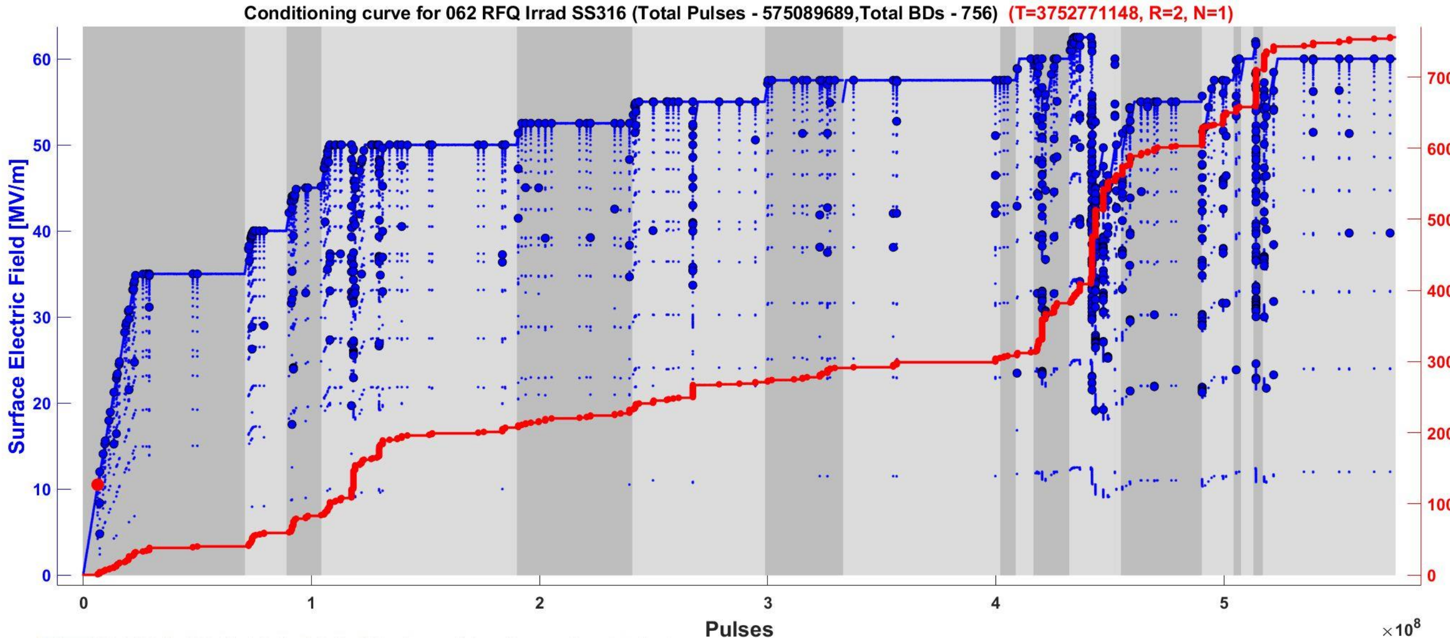


SS316 electrode non-irradiated

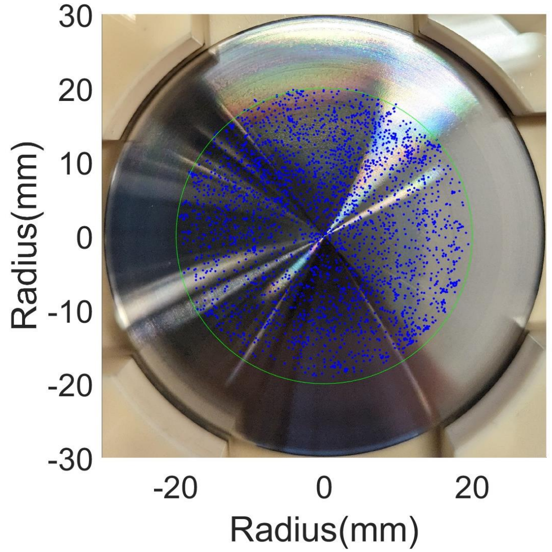


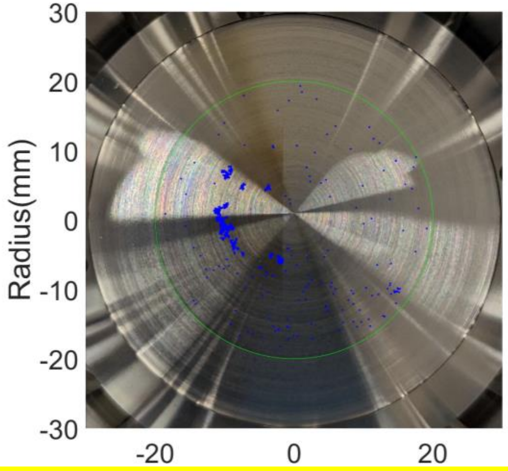




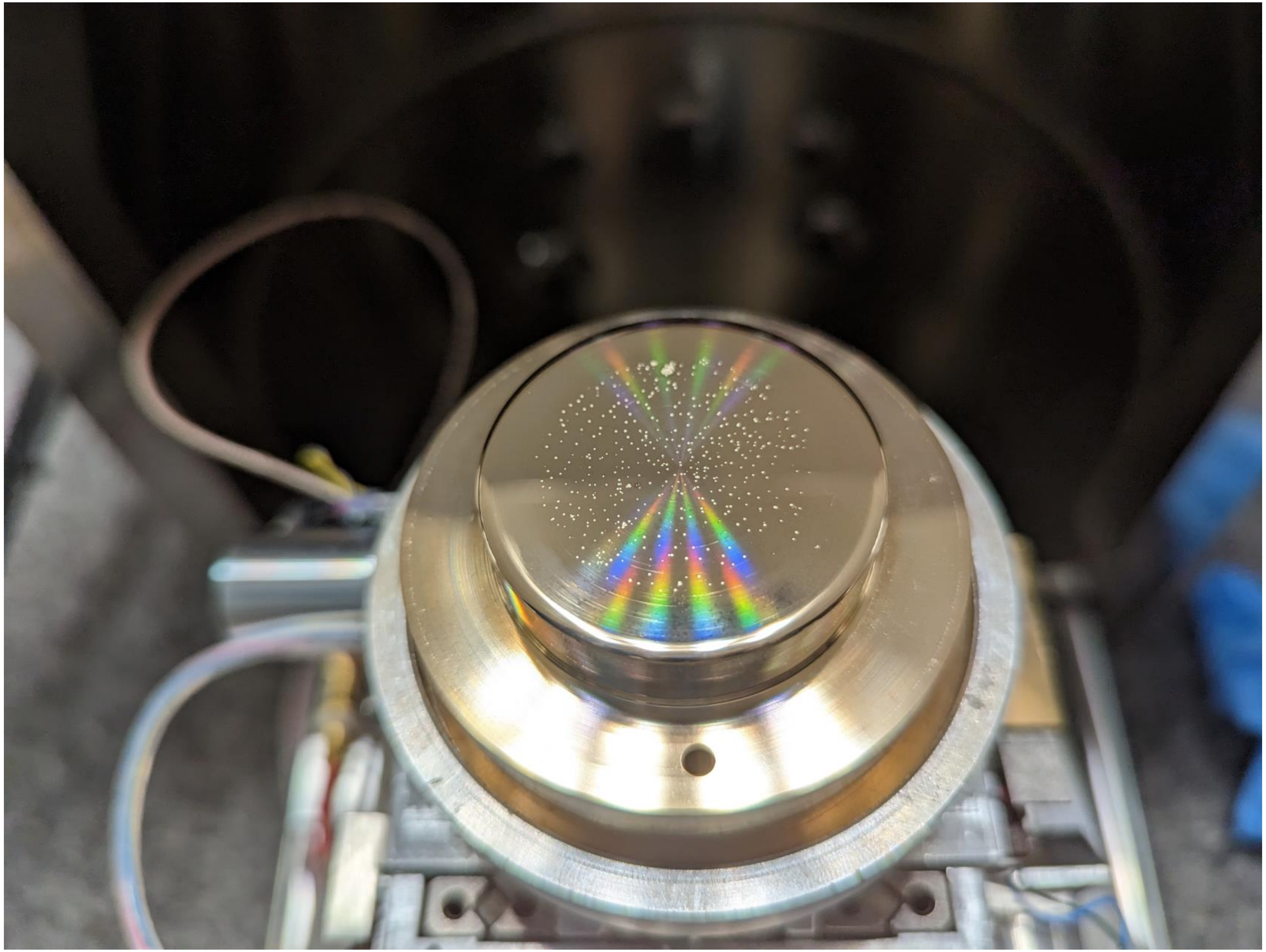


Stainless Steel electrodes non-irradiated (last 12 runs).
Maximum field 120 MV/m.

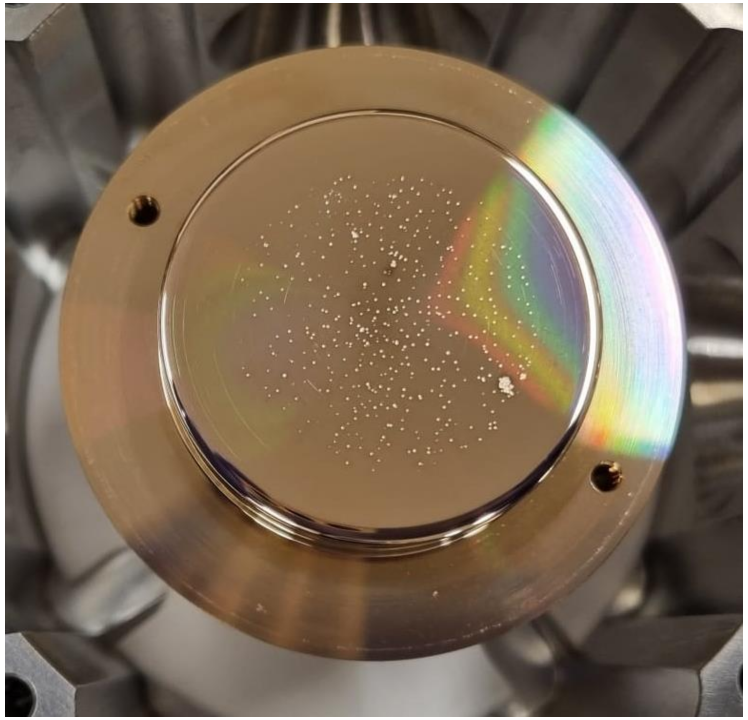




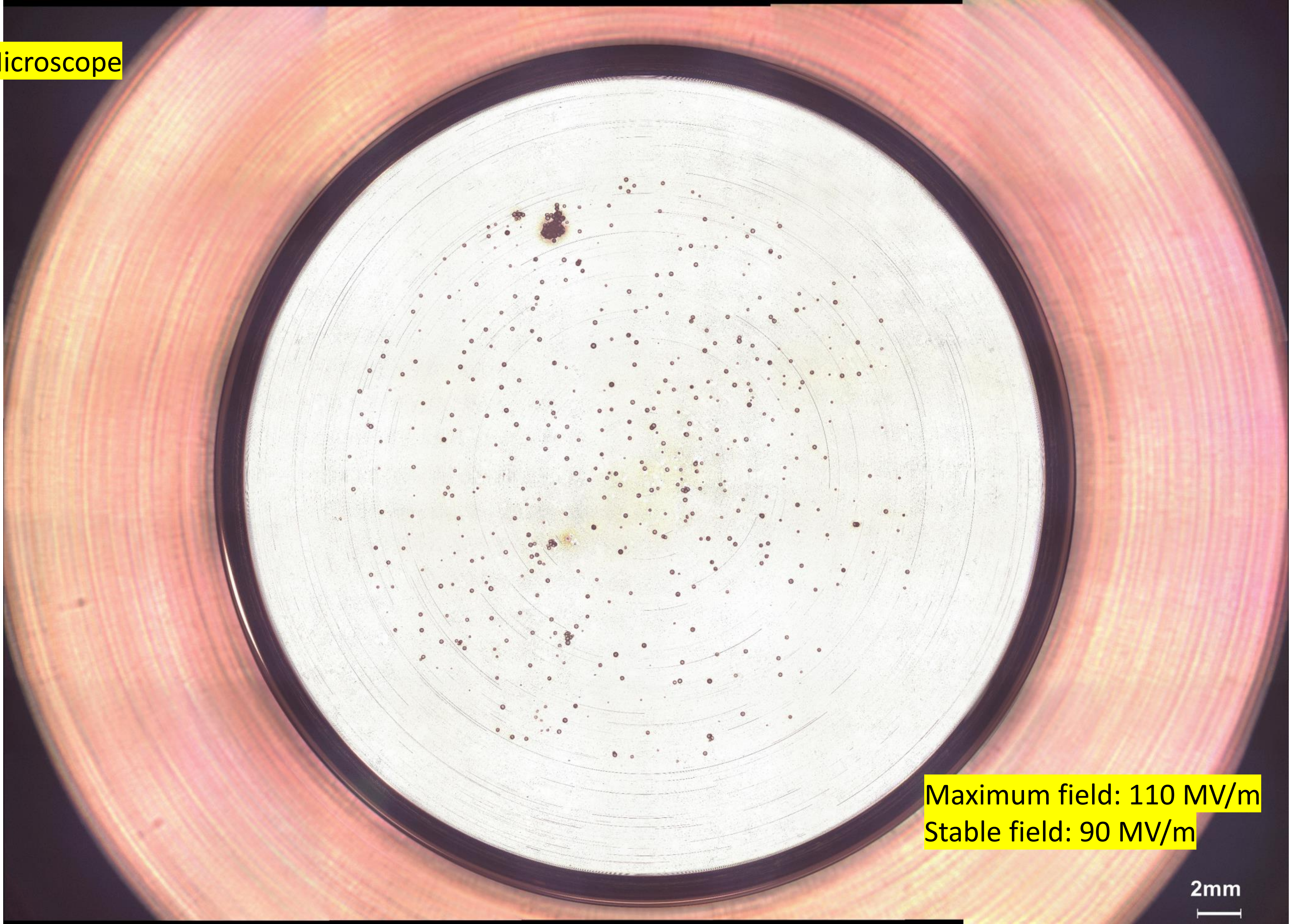
Stainless Steel electrodes irradiated
Maximum field 62 MV/m.



CuBe2 electrodes
(non-irradiated and LES tested)

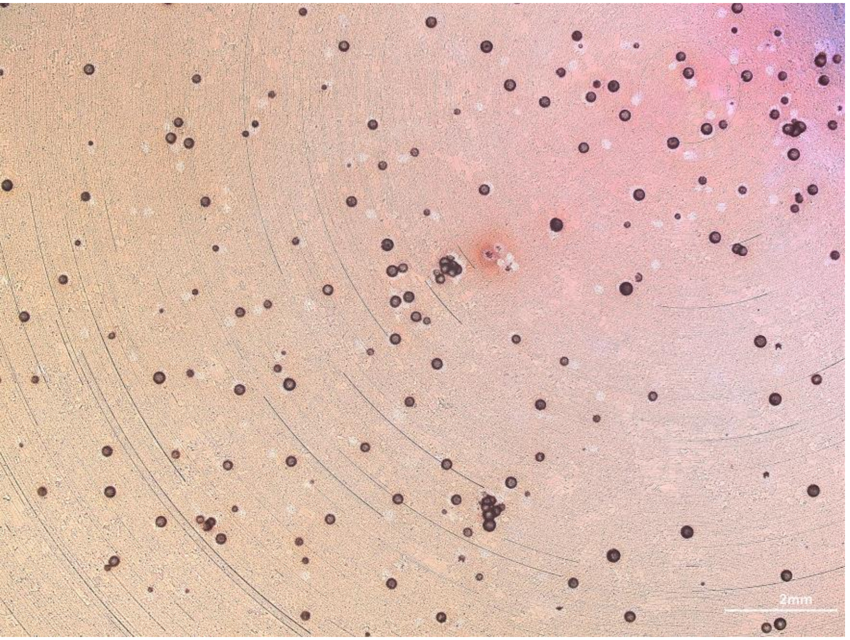


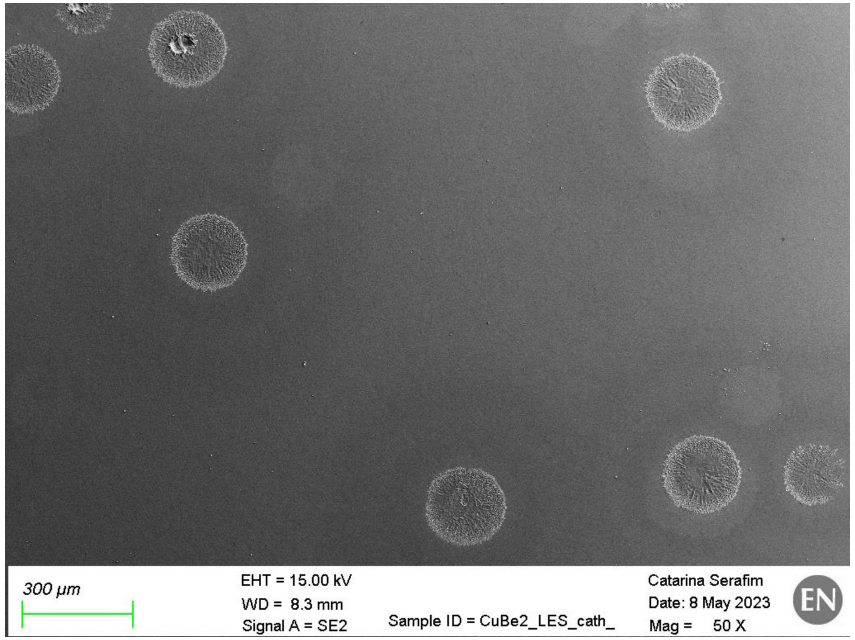
Optical Microscope

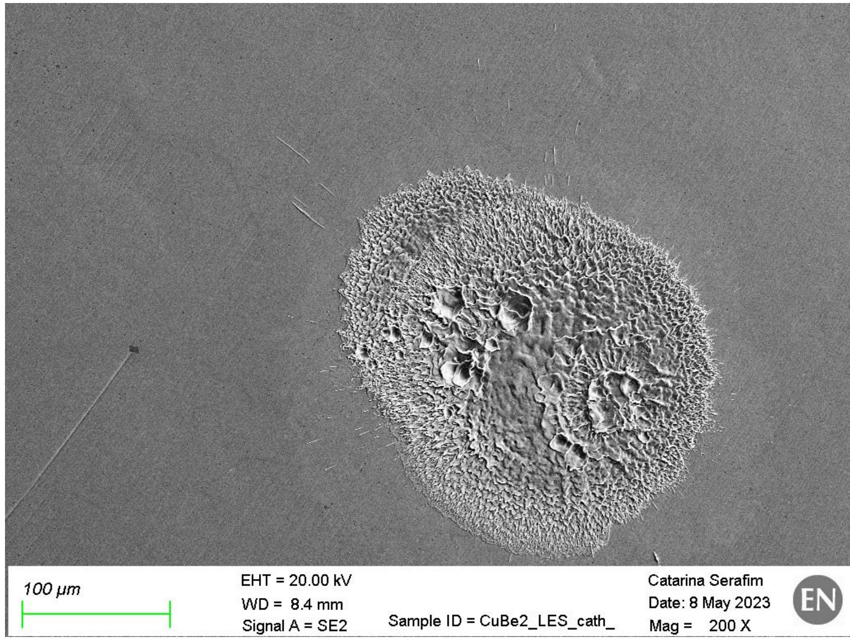


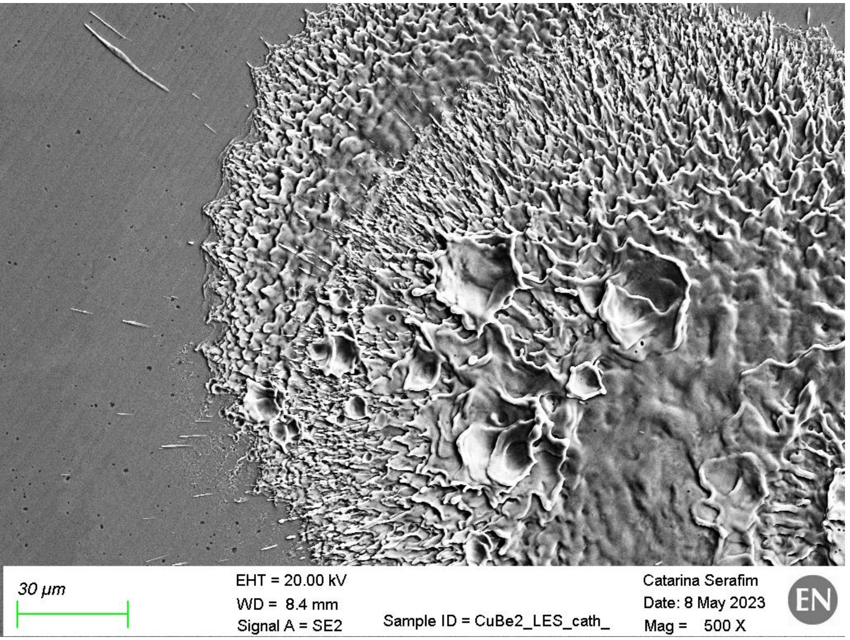
Maximum field: 110 MV/m
Stable field: 90 MV/m

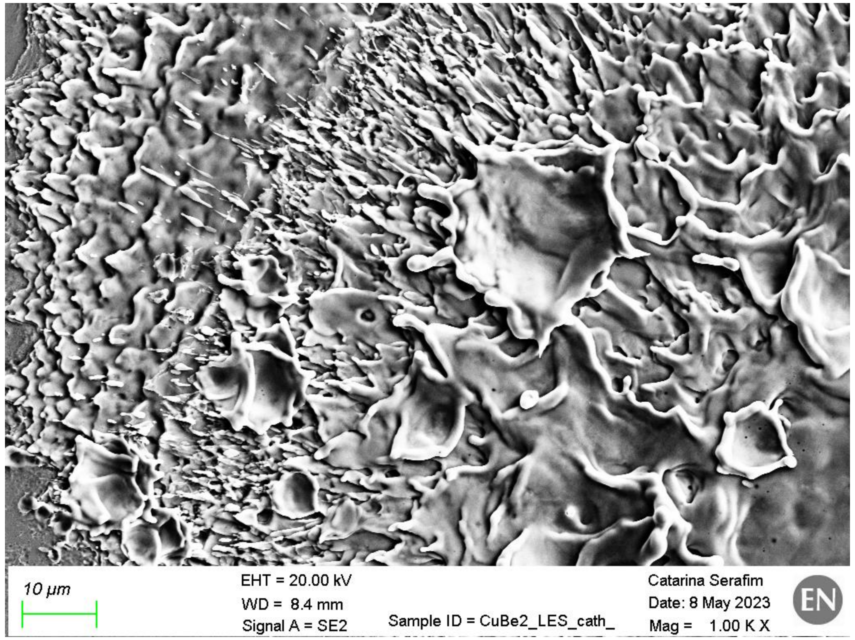
SEM

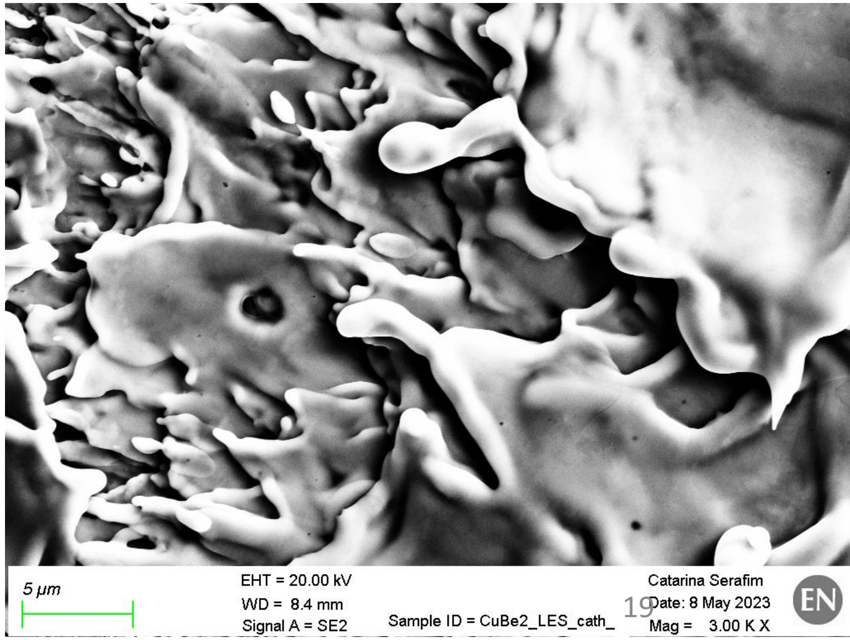

2mm

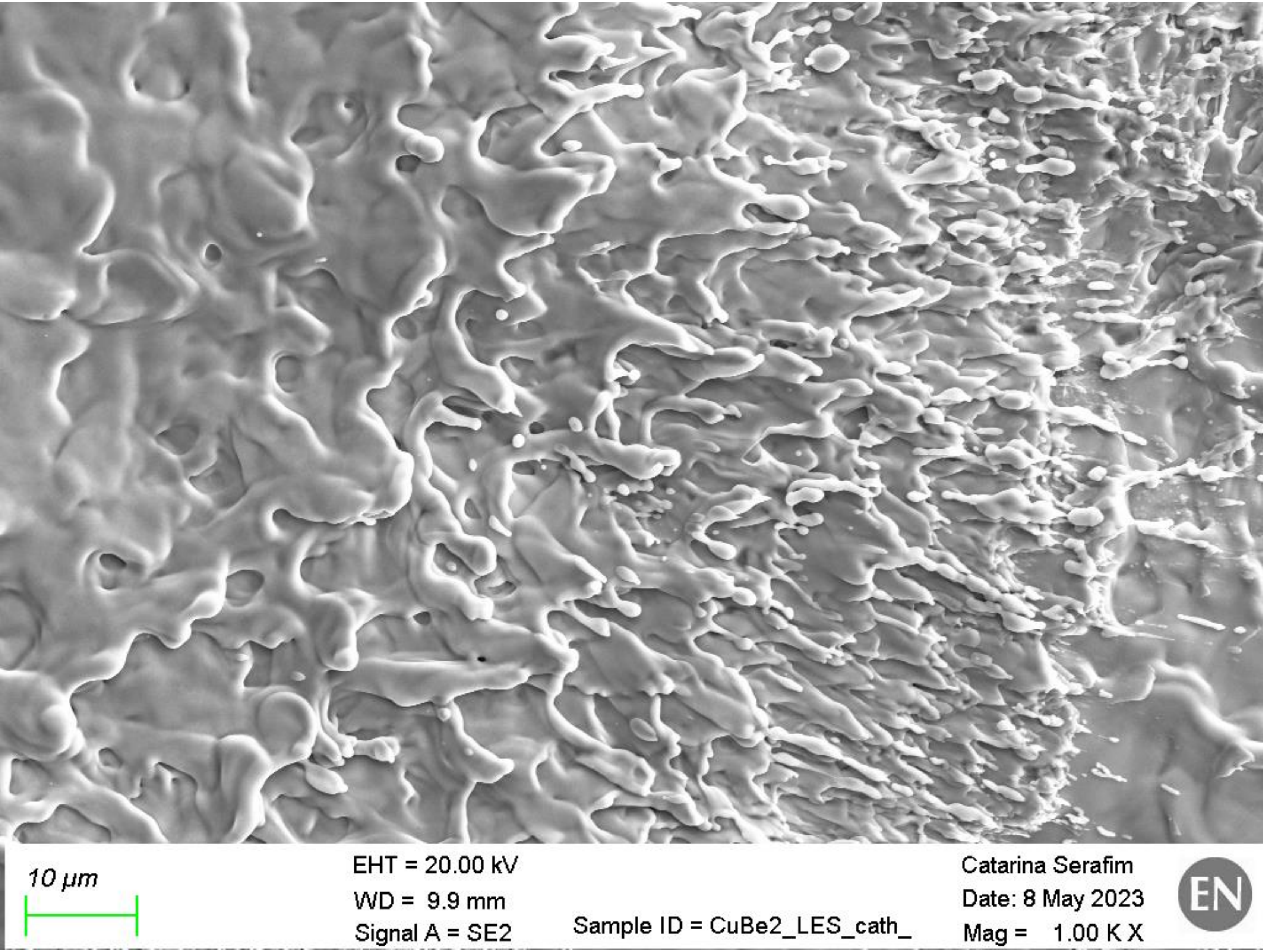

300 µm
EHT = 15.00 kV
WD = 8.3 mm
Signal A = SE2
Sample ID = CuBe2_LES_cath_
Catarina Serafim
Date: 8 May 2023
Mag = 50 X
EN

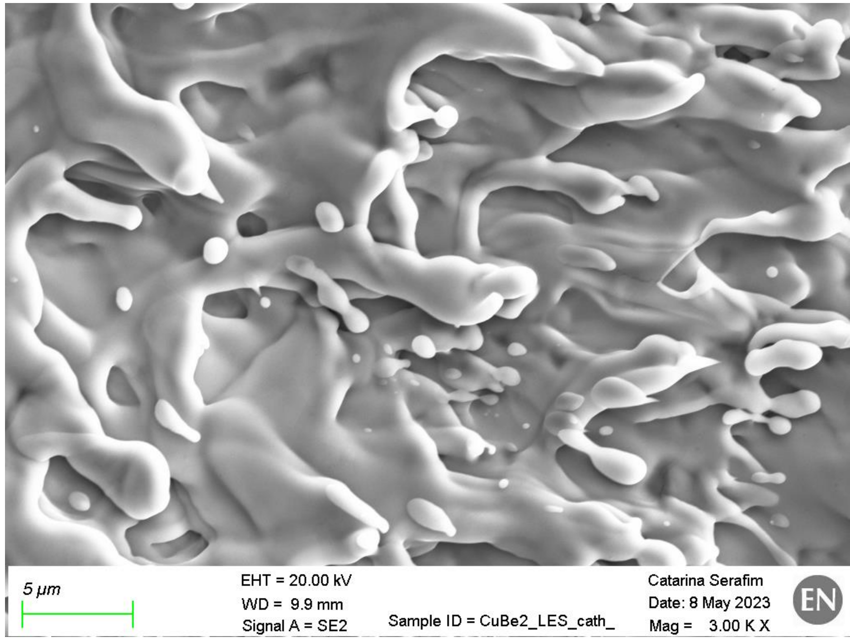

100 µm
EHT = 20.00 kV
WD = 8.4 mm
Signal A = SE2
Sample ID = CuBe2_LES_cath_
Catarina Serafim
Date: 8 May 2023
Mag = 200 X
EN

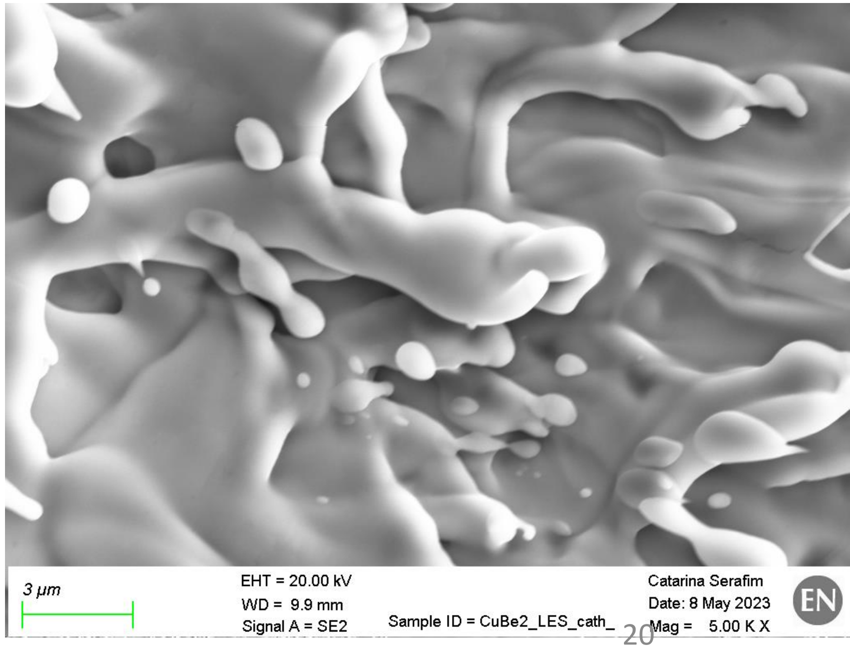

30 µm
EHT = 20.00 kV
WD = 8.4 mm
Signal A = SE2
Sample ID = CuBe2_LES_cath_
Catarina Serafim
Date: 8 May 2023
Mag = 500 X
EN


10 µm
EHT = 20.00 kV
WD = 8.4 mm
Signal A = SE2
Sample ID = CuBe2_LES_cath_
Catarina Serafim
Date: 8 May 2023
Mag = 1.00 K X
EN

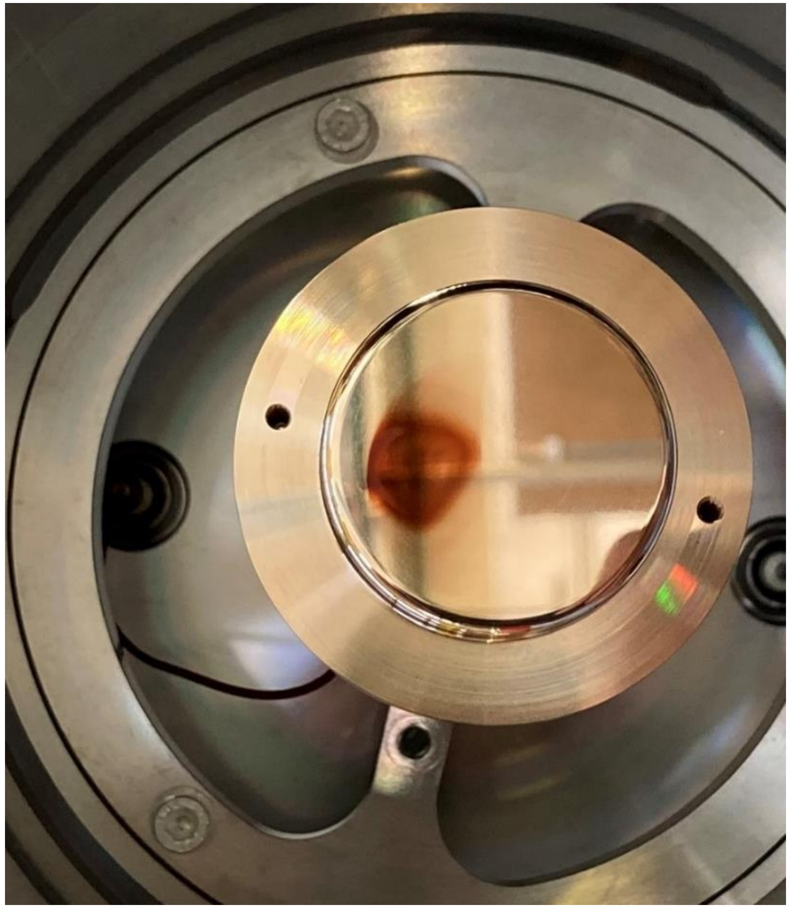

5 µm
EHT = 20.00 kV
WD = 8.4 mm
Signal A = SE2
Sample ID = CuBe2_LES_cath_
Catarina Serafim
Date: 8 May 2023
Mag = 3.00 K X
EN


10 µm
EHT = 20.00 kV
WD = 9.9 mm
Signal A = SE2
Sample ID = CuBe2_LES_cath_
Catarina Serafim
Date: 8 May 2023
Mag = 1.00 K X

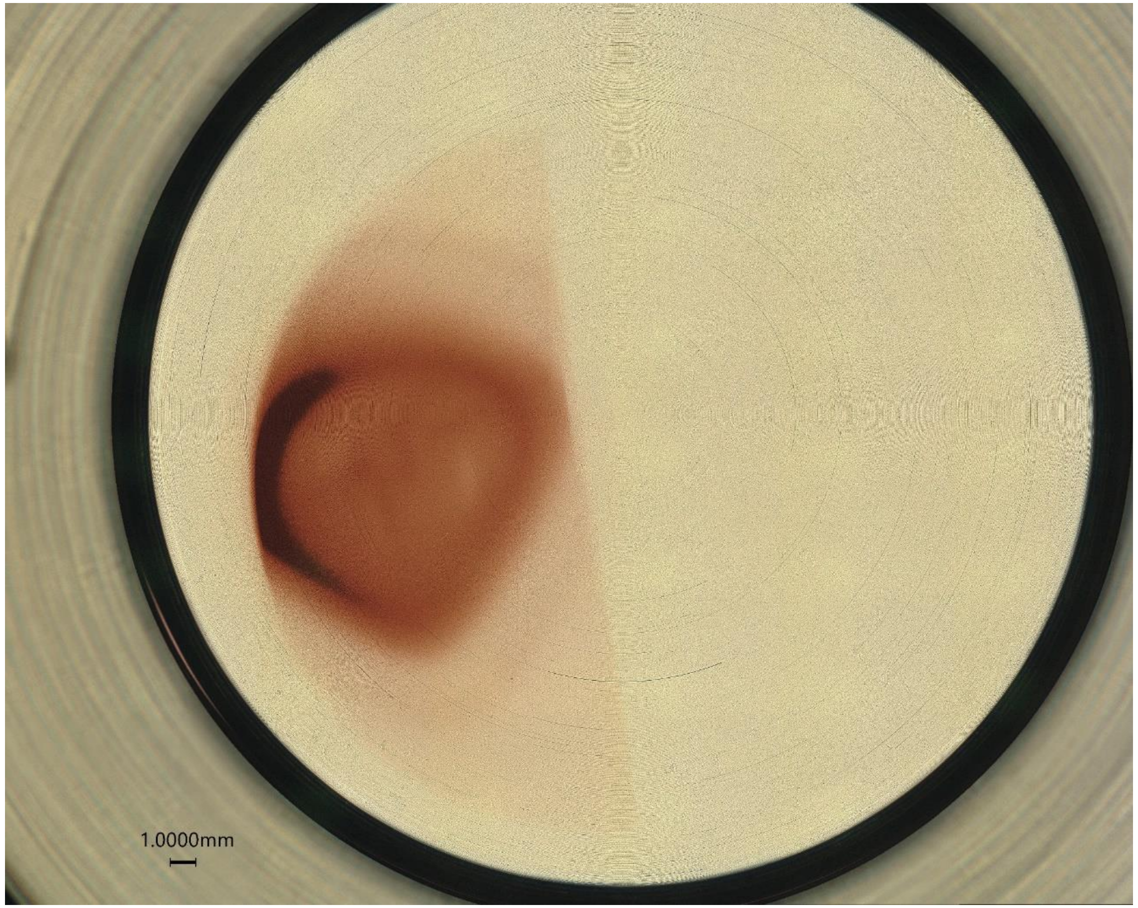

5 µm
EHT = 20.00 kV
WD = 9.9 mm
Signal A = SE2
Sample ID = CuBe2_LES_cath_
Catarina Serafim
Date: 8 May 2023
Mag = 3.00 K X


3 µm
EHT = 20.00 kV
WD = 9.9 mm
Signal A = SE2
Sample ID = CuBe2_LES_cath_
Catarina Serafim
Date: 8 May 2023
Mag = 5.00 K X

CuBe2 cathode – irradiated



Dismounting of CuBe cathode after irradiation

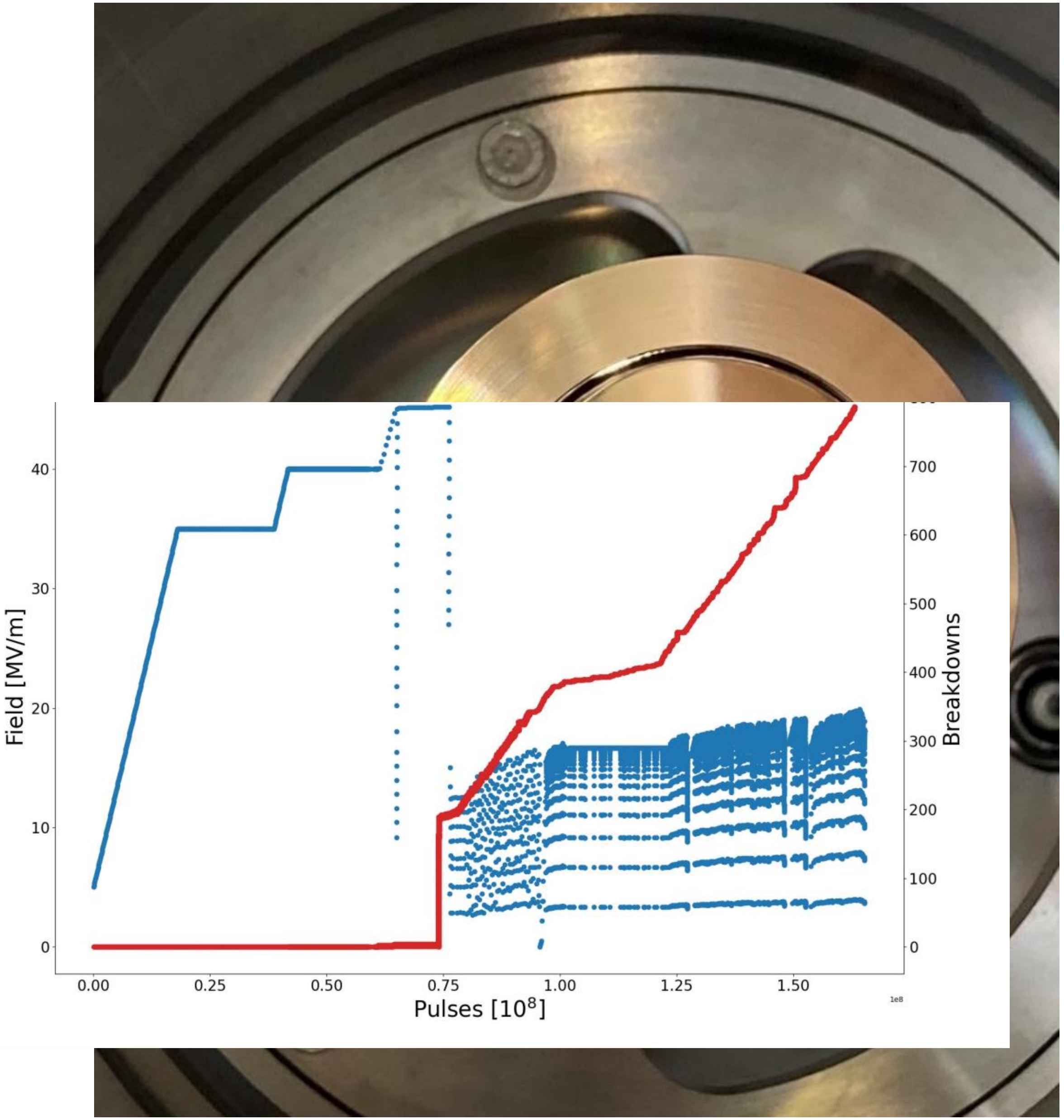


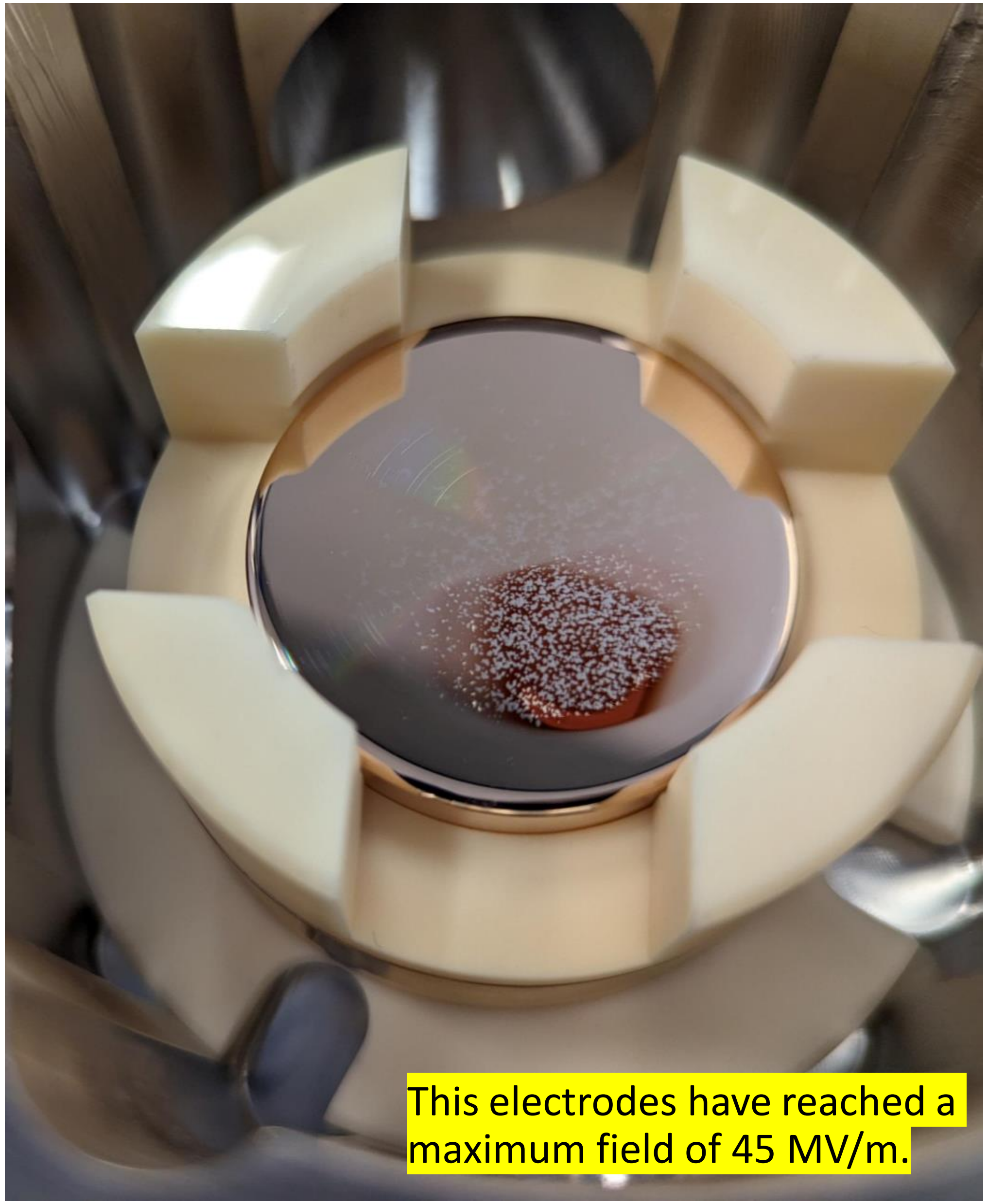
Optical imaging of the surface using lens 80x.


CuBe2 cathode – after irradiation


CuBe2 cathode irradiated after LES testing
This electrodes have reached a maximum field of 45 MV/m.

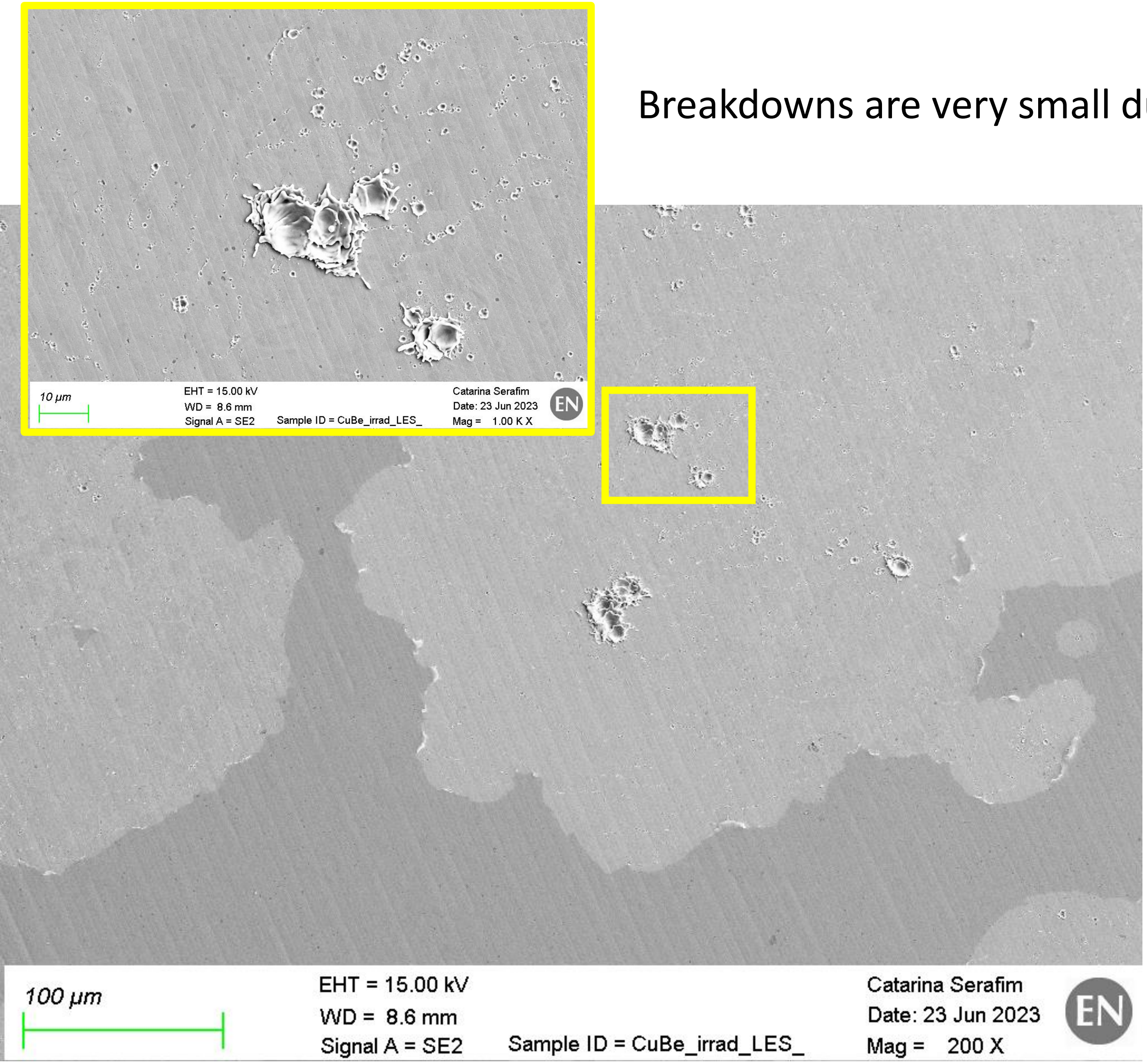
CuBe irradiated + LES

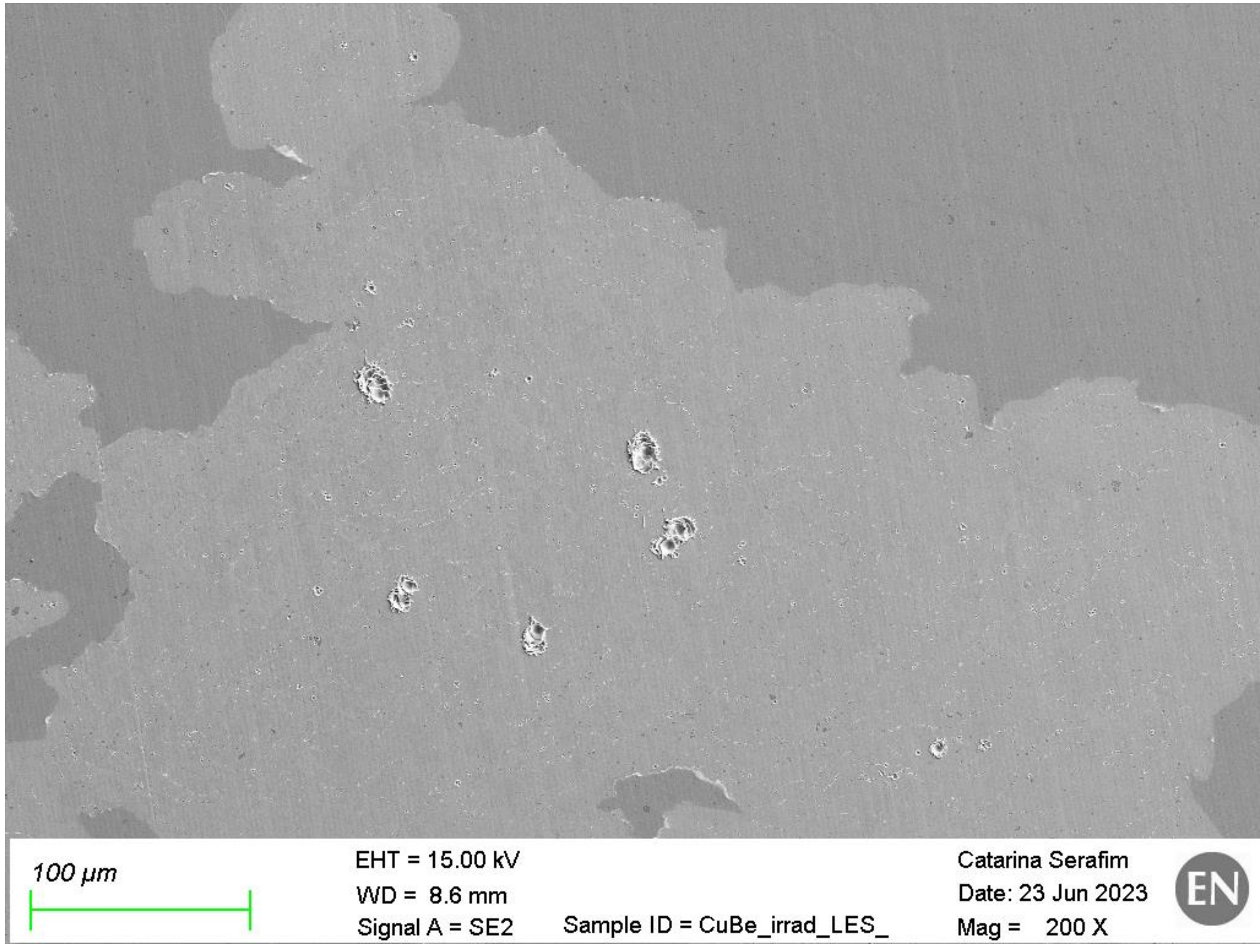




This electrodes have reached a maximum field of 45 MV/m.

Breakdowns are very small due to the low field achieved.





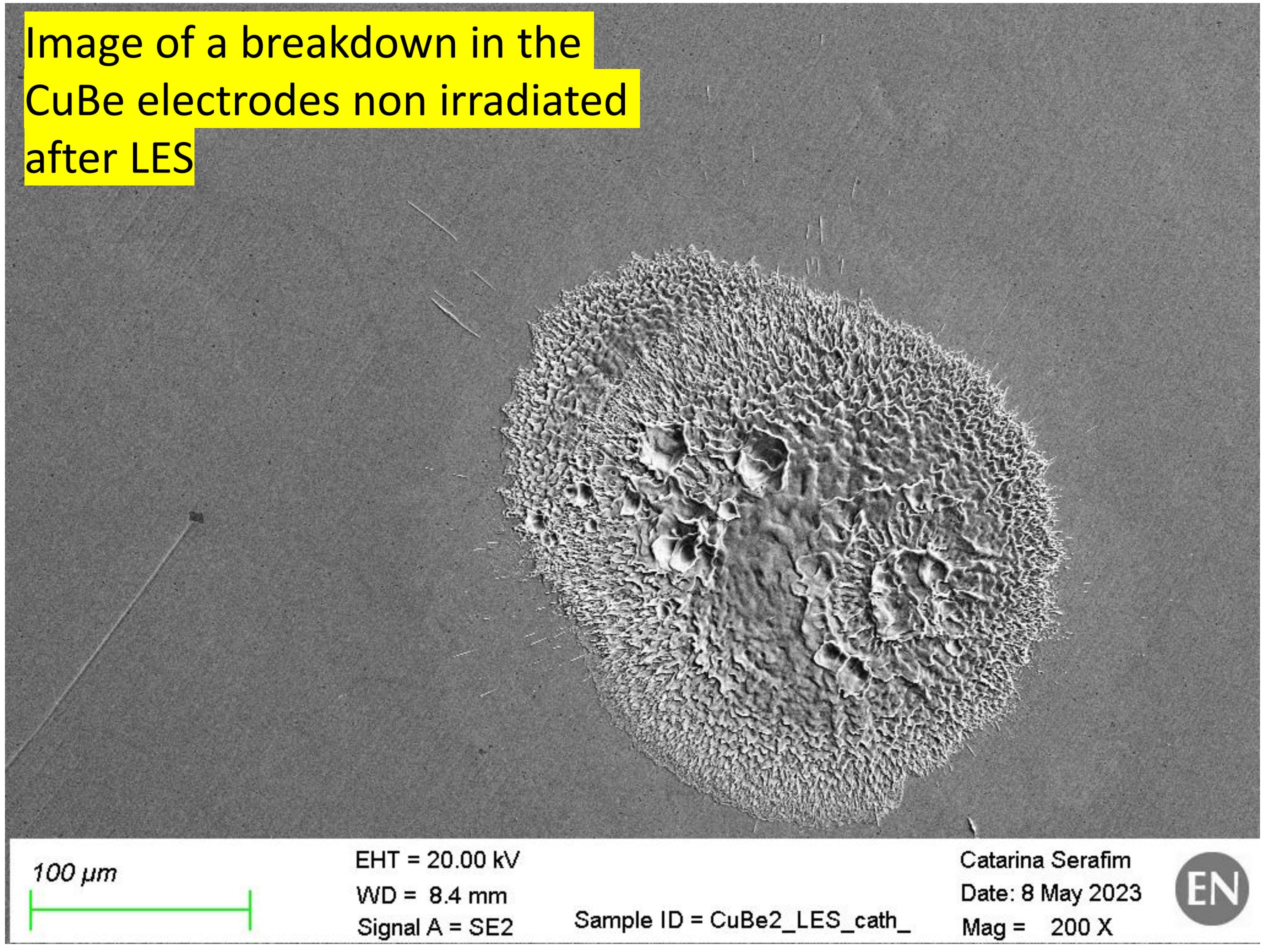
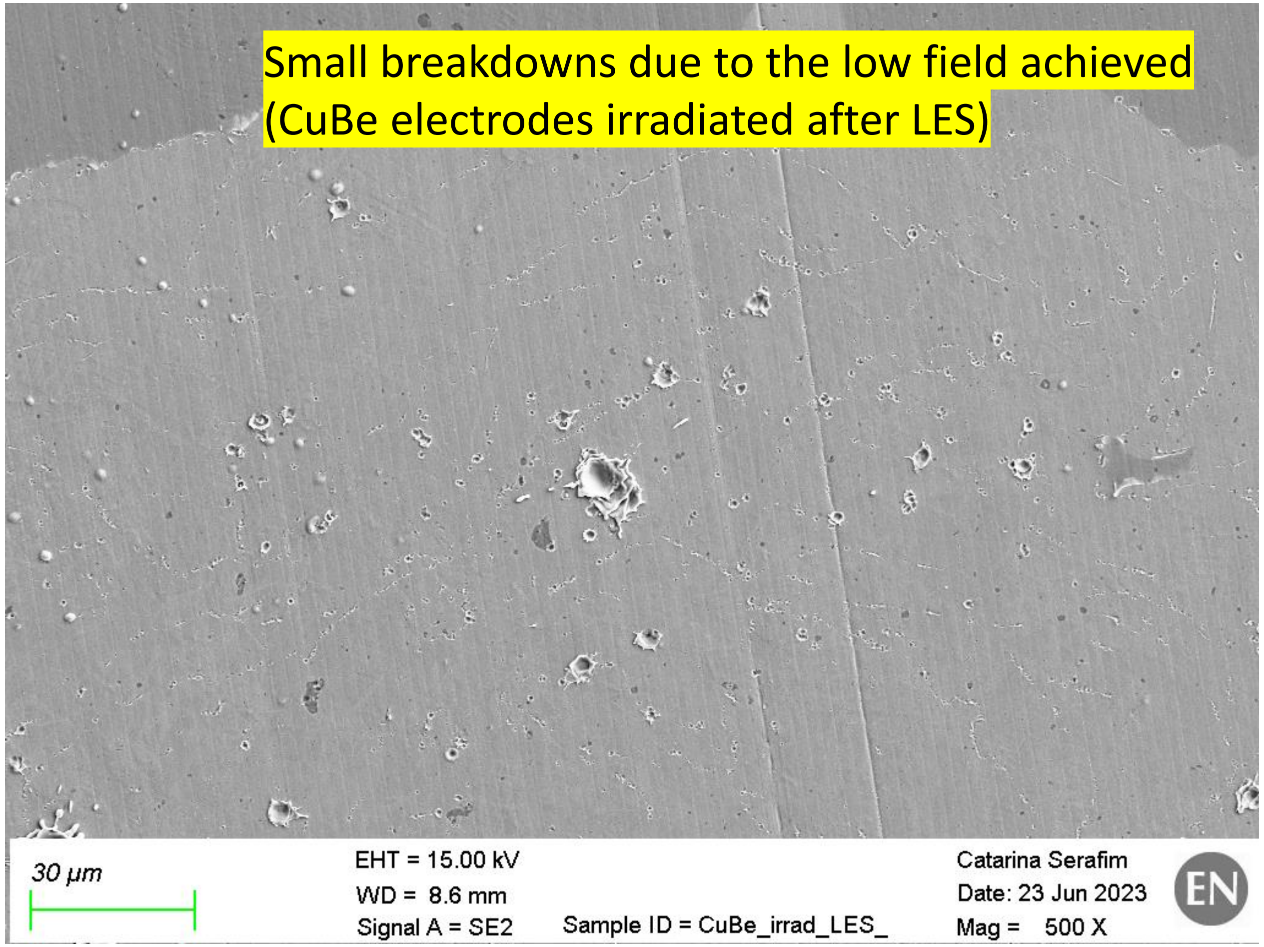


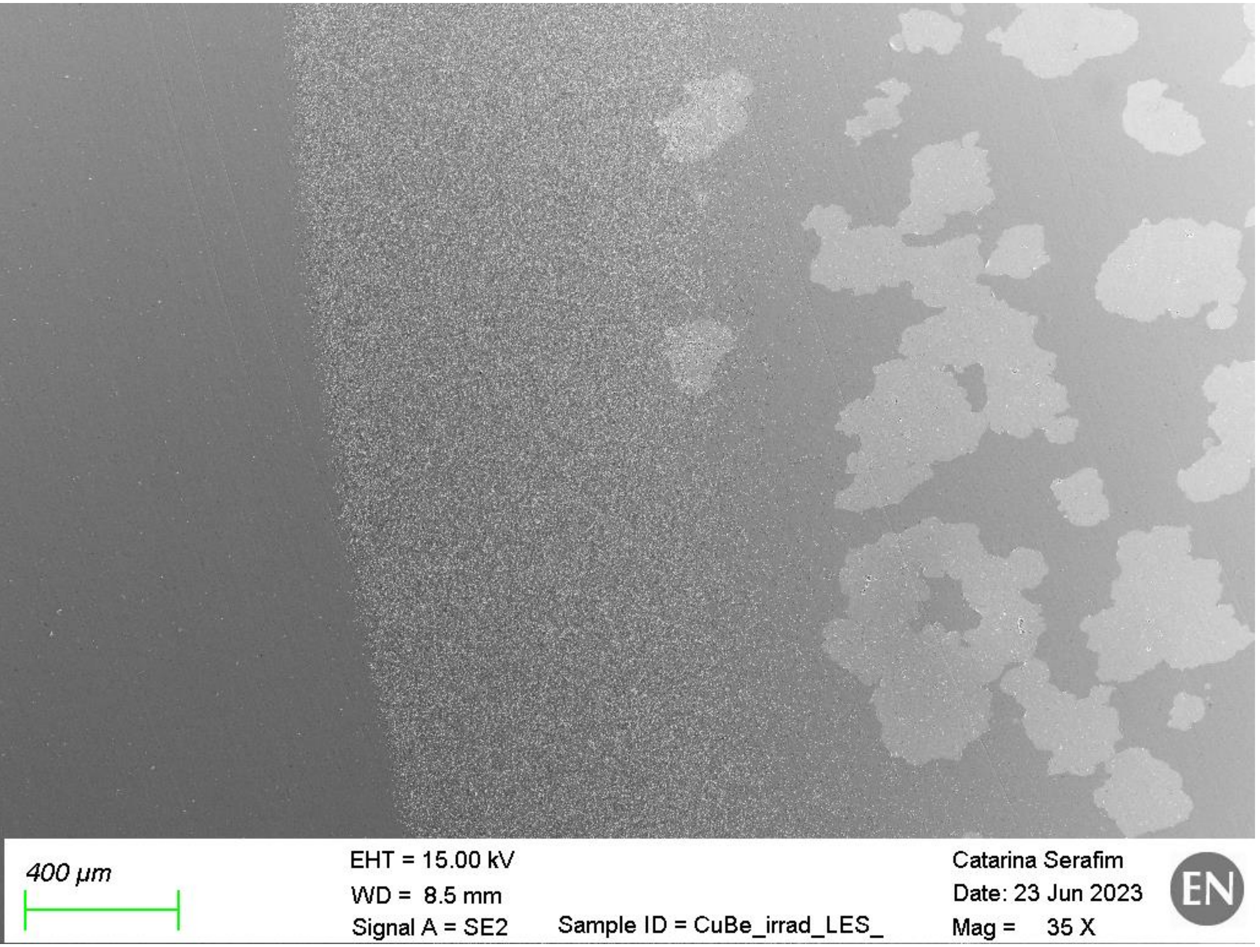
Image of a breakdown in the
CuBe electrodes non irradiated
after LES
100 µm
EHT = 20.00 kV
WD = 8.4 mm
Signal A = SE2
Sample ID = CuBe2_LES_cath_
Catarina Serafim
Date: 8 May 2023
Mag = 200 X

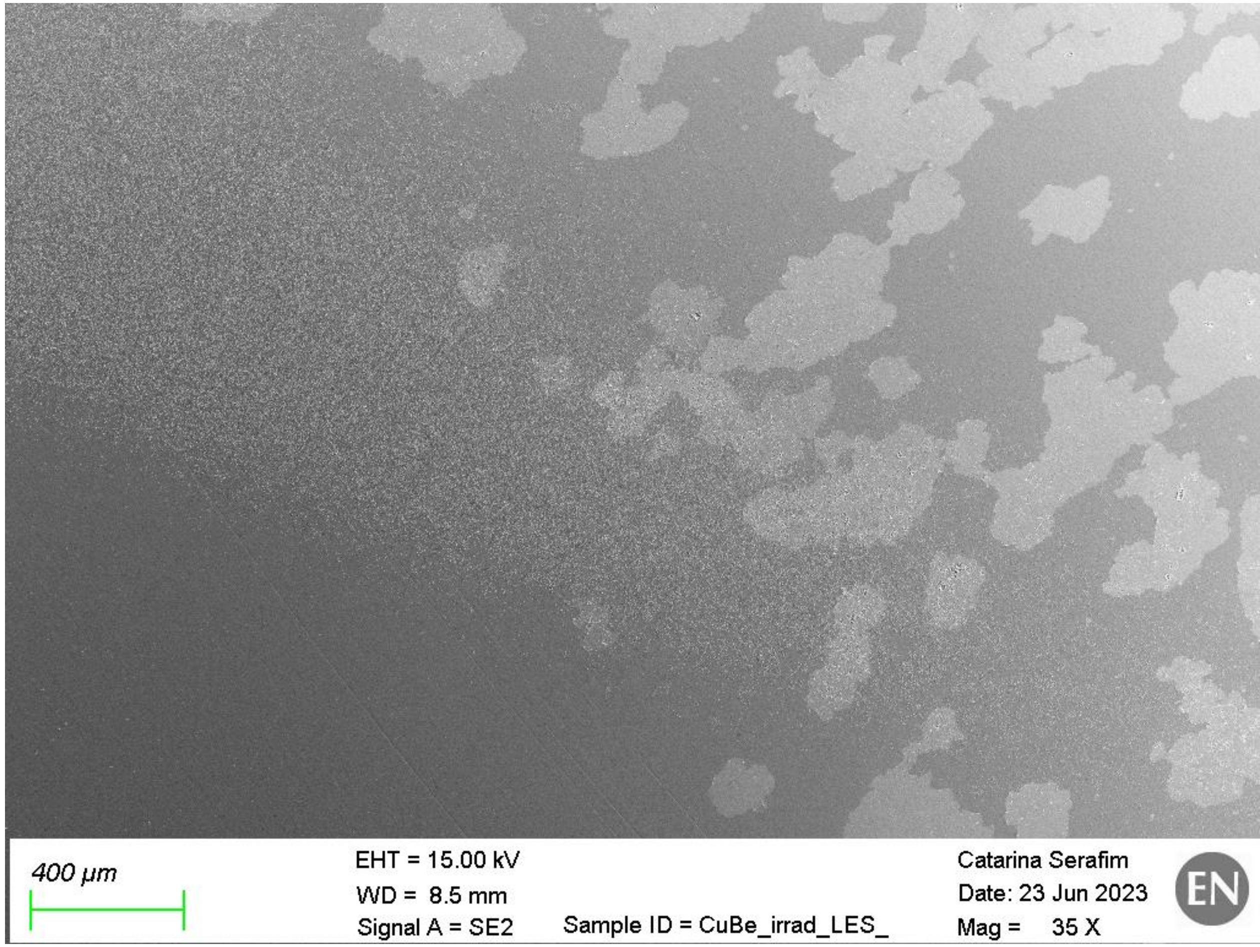

Small breakdowns due to the low field achieved
(CuBe electrodes irradiated after LES)
30 µm
EHT = 15.00 kV
WD = 8.6 mm
Signal A = SE2
Sample ID = CuBe_irrad_LES_
Catarina Serafim
Date: 23 Jun 2023
Mag = 500 X

Irradiated cathode, submitted to high pulsing test.

Similar to previous irradiations, we have observed a carbon content inside the irradiated zone.

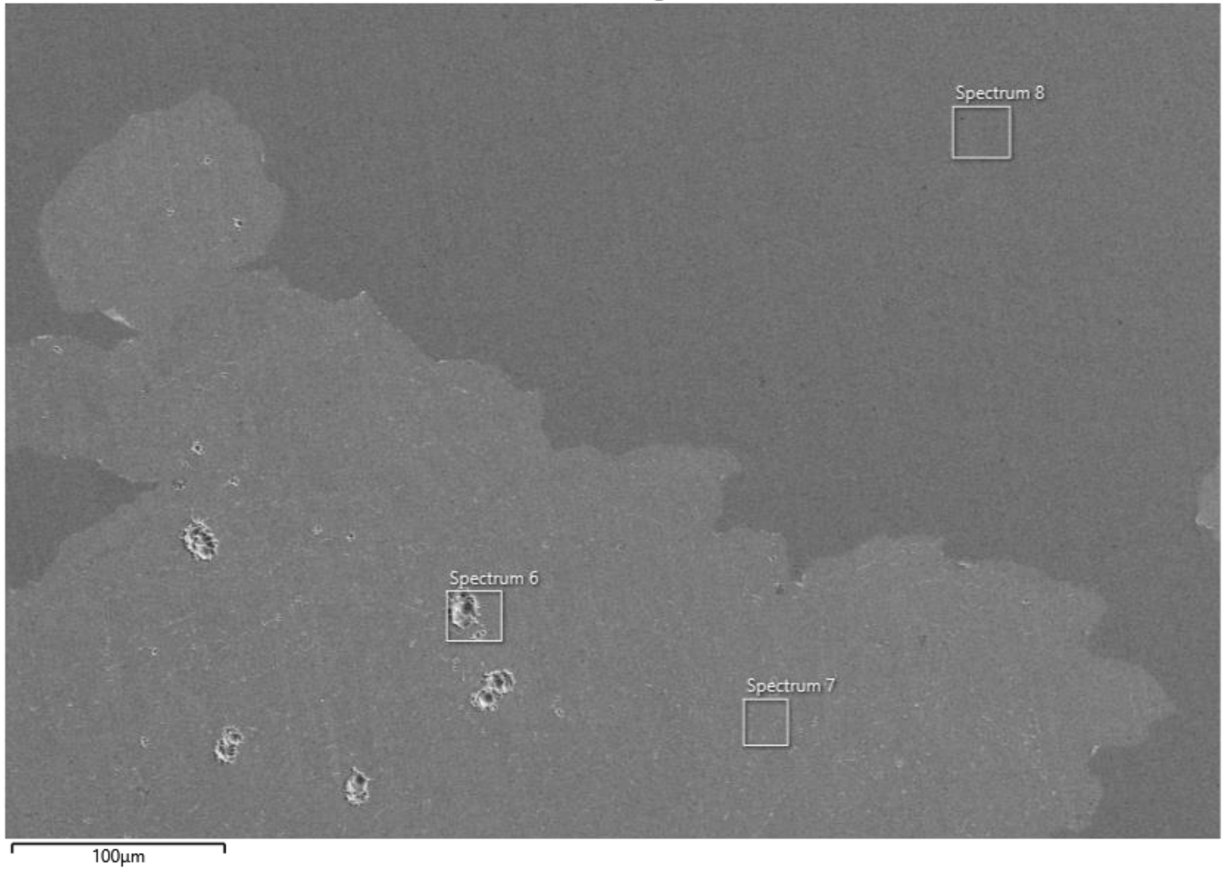
In the irradiated zone, exfoliation of this carbon layer was observed (white spots).





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

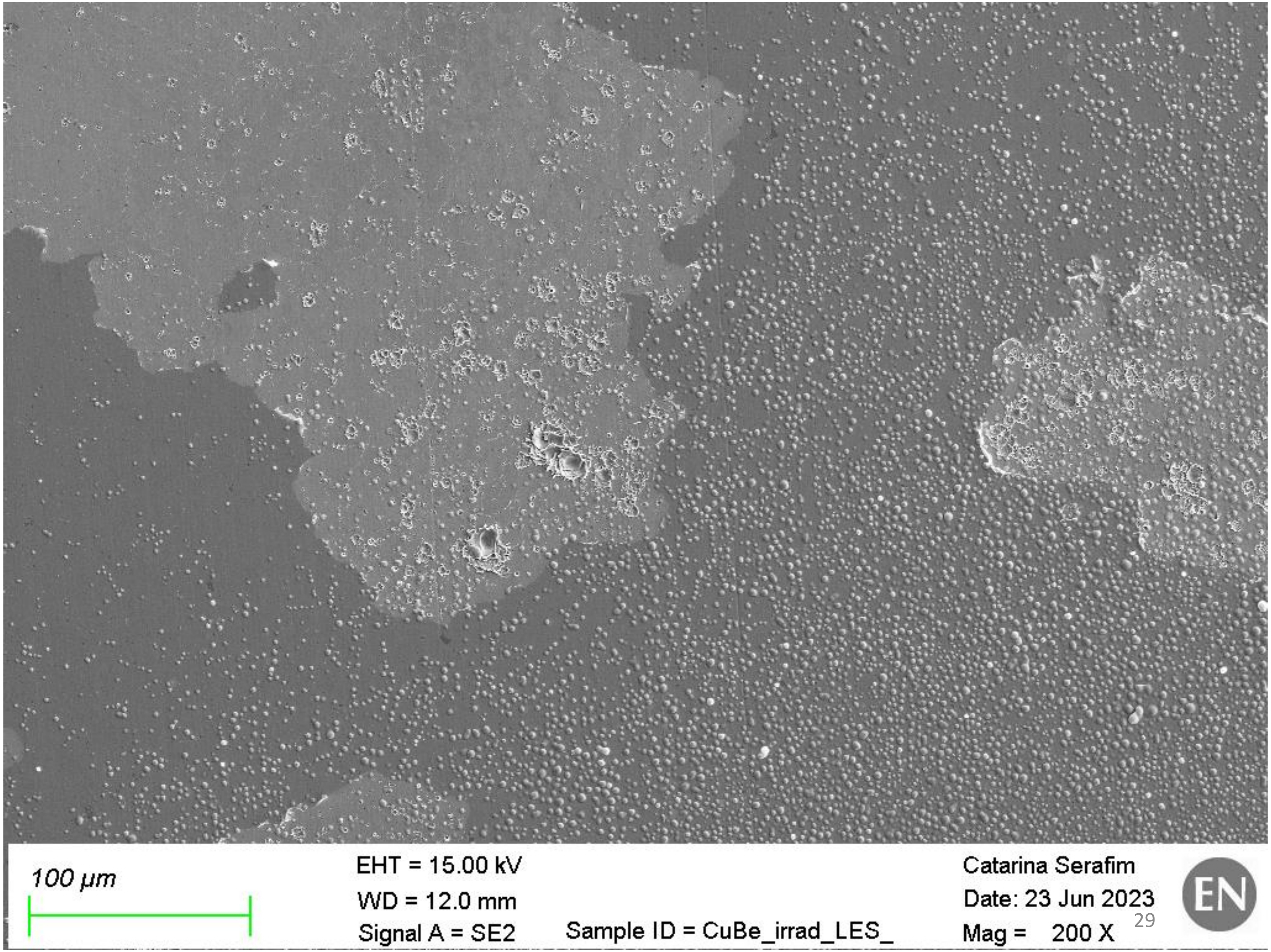
Element	Line Type	Wt%
C	K series	1.38
Cu	L series	98.62

Spectrum 5

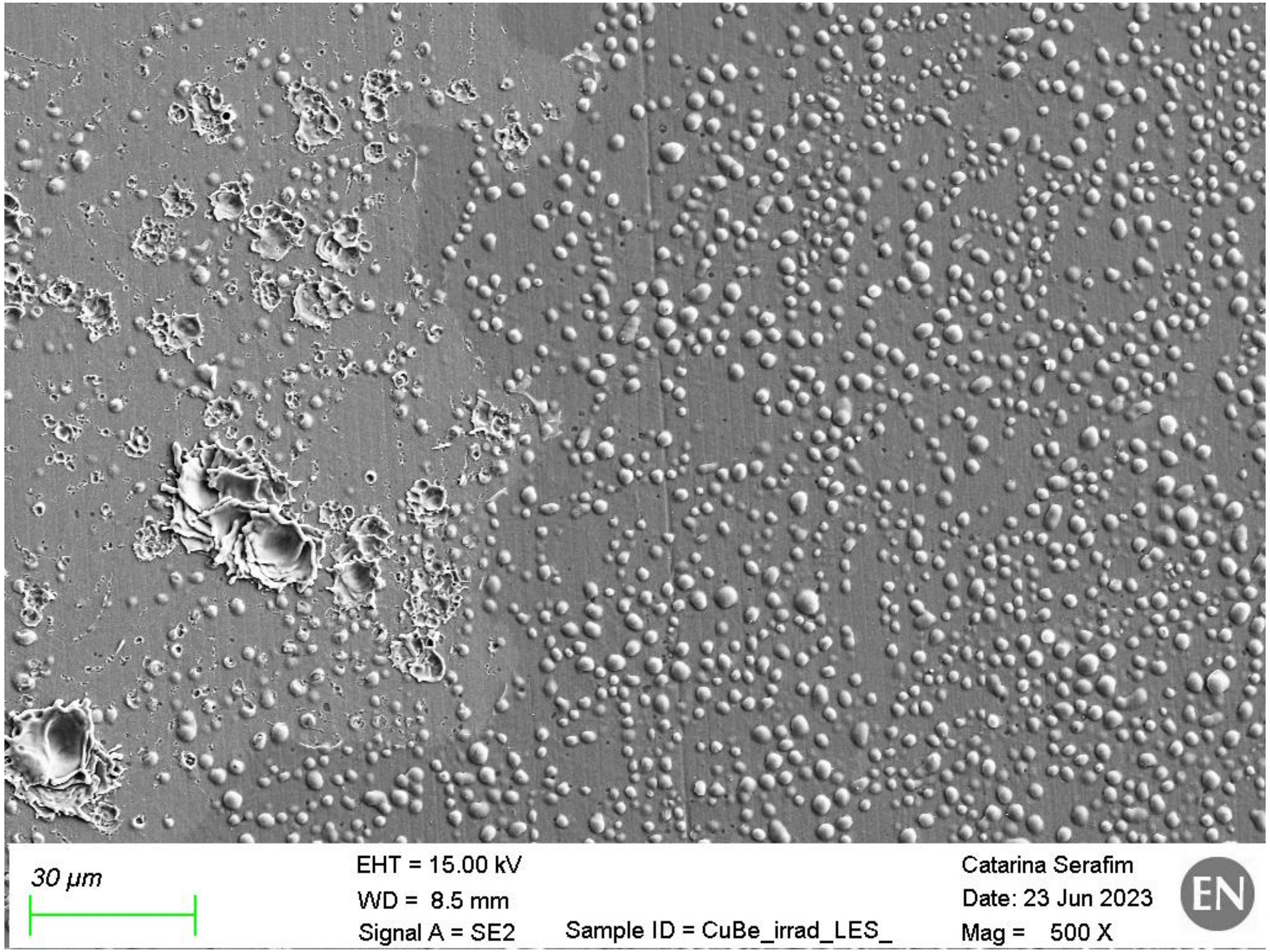
Element	Line Type	Wt%
C	K series	11.04
Cu	L series	88.96

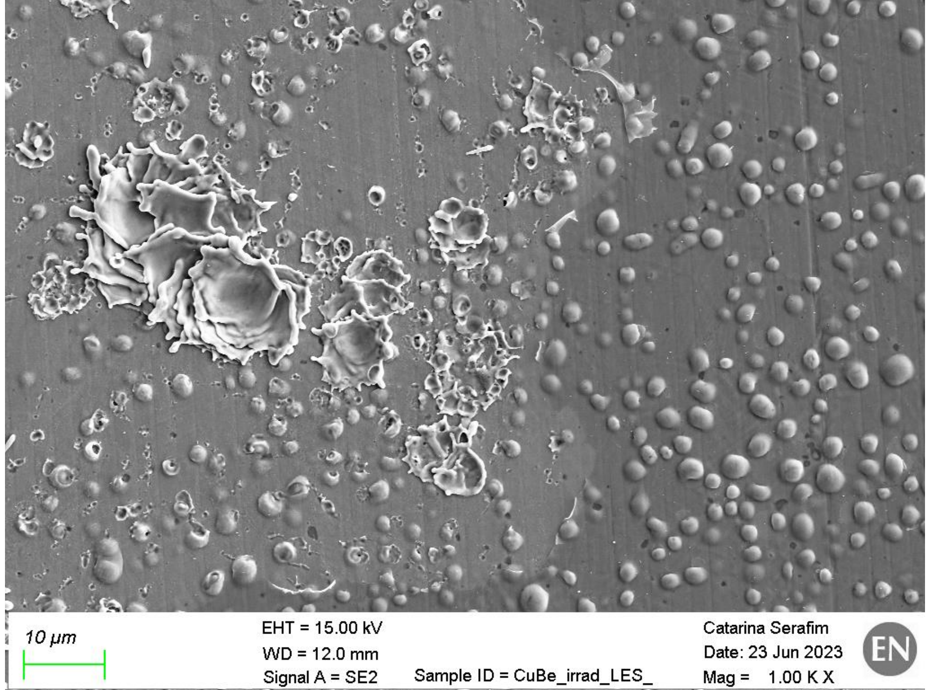
We have observed that the breakdowns are only concentrated inside the exfoliated zones.

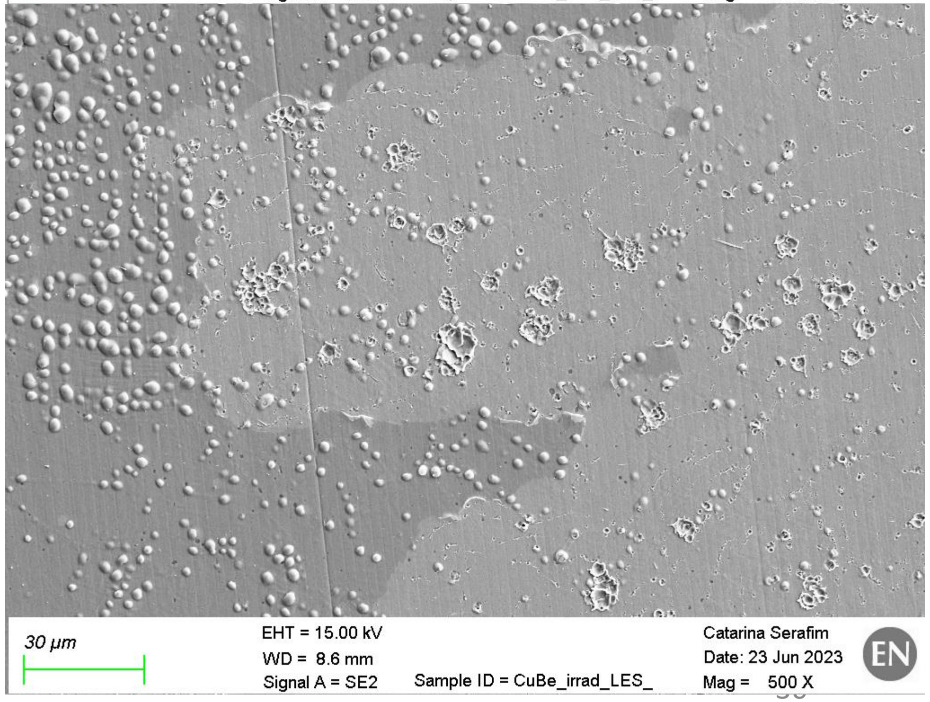
The zones without those exfoliations, i.e. with a protective carbon layer, are free from breakdowns, even in the zones with high density of blisters.



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

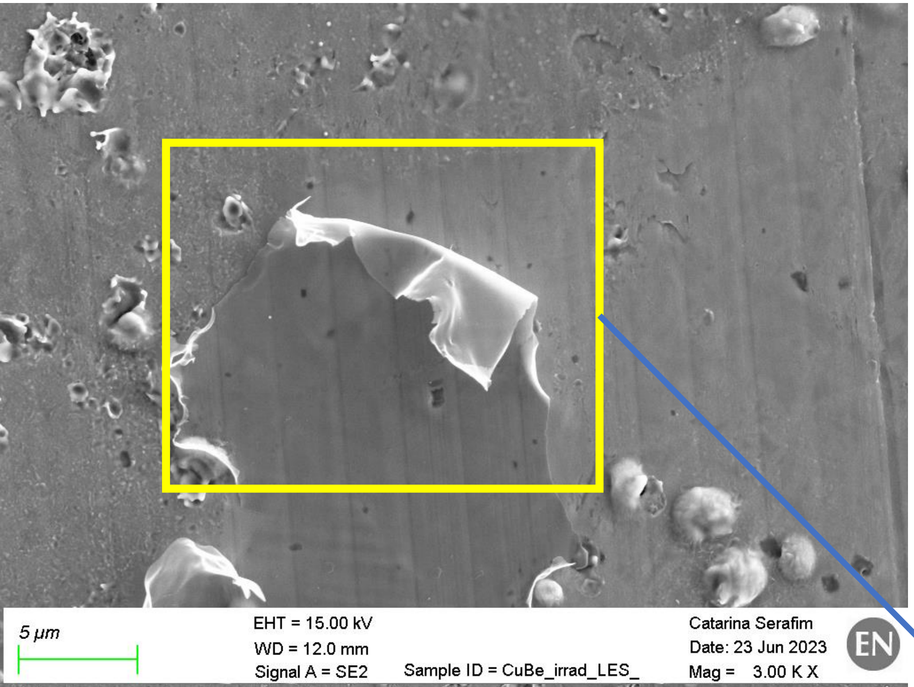




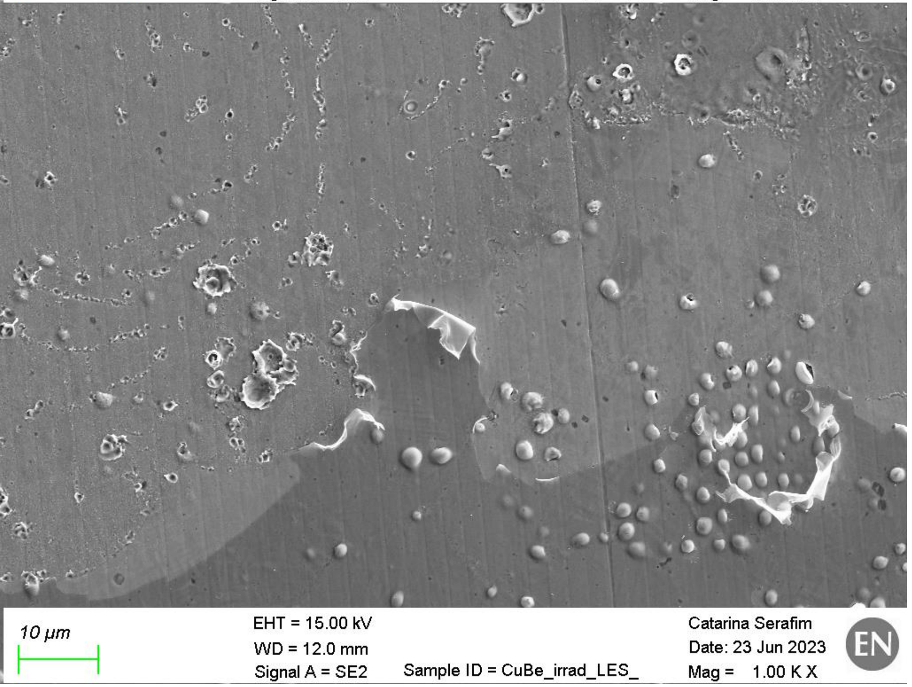


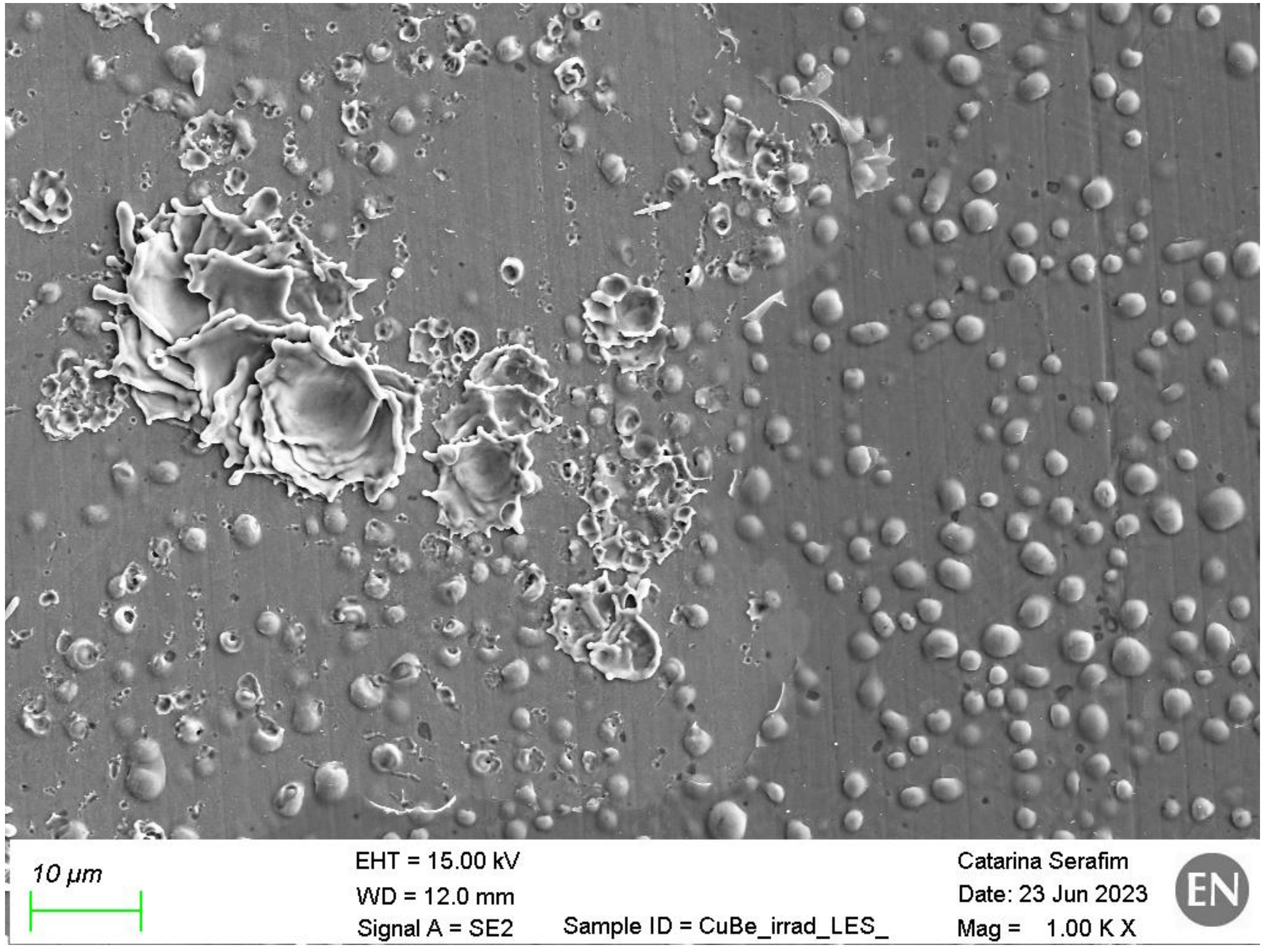
Is the carbon preventing the electrodes from reaching a high field?

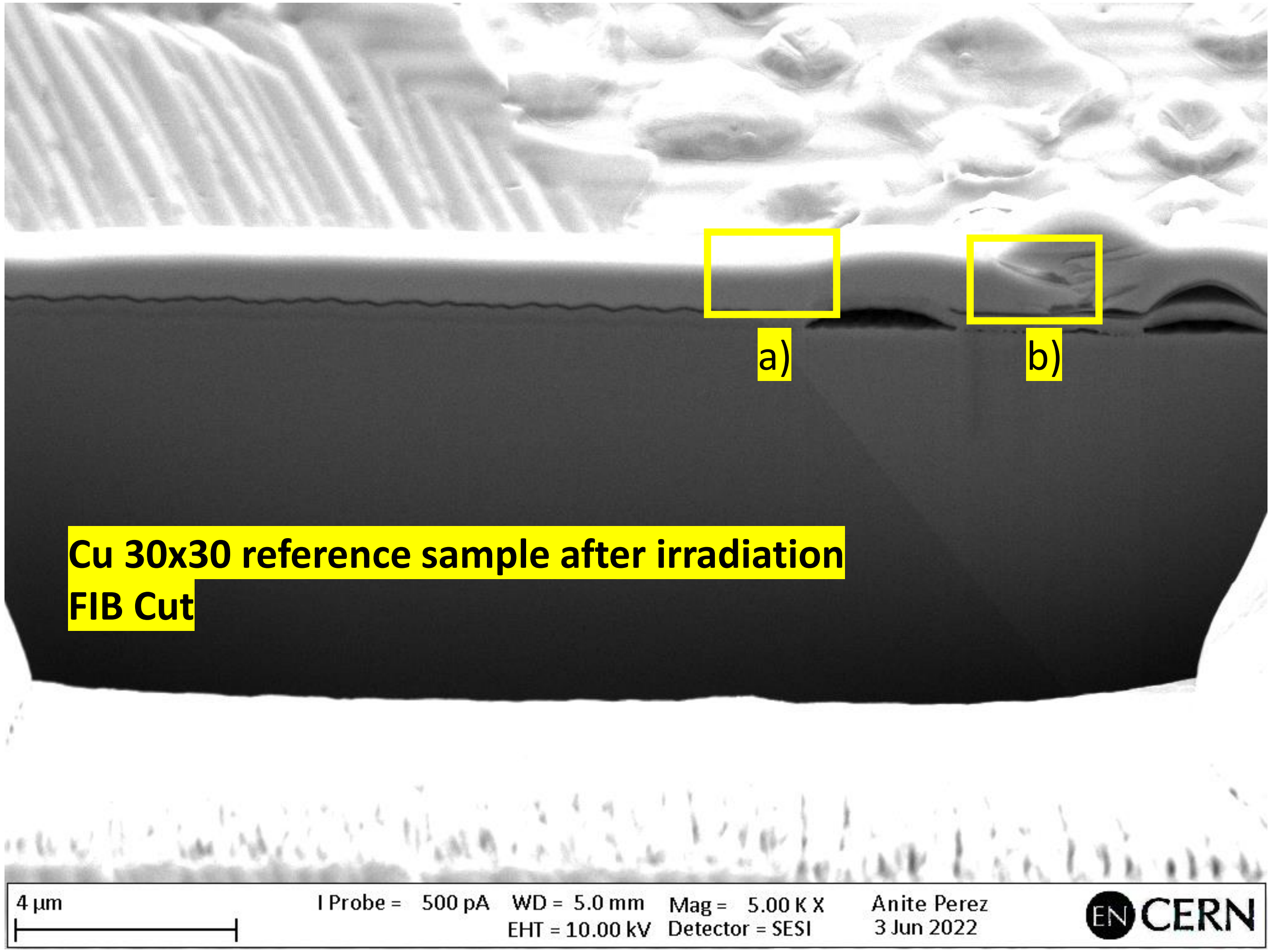
Plan for Plasma cleaning treatment
+ Repeat LES testing

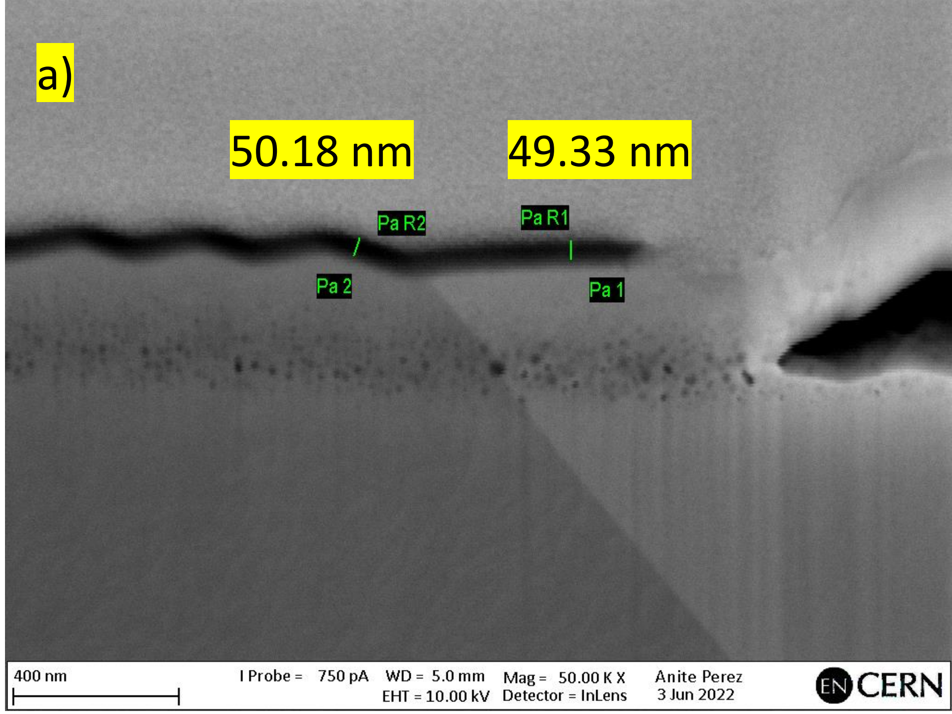


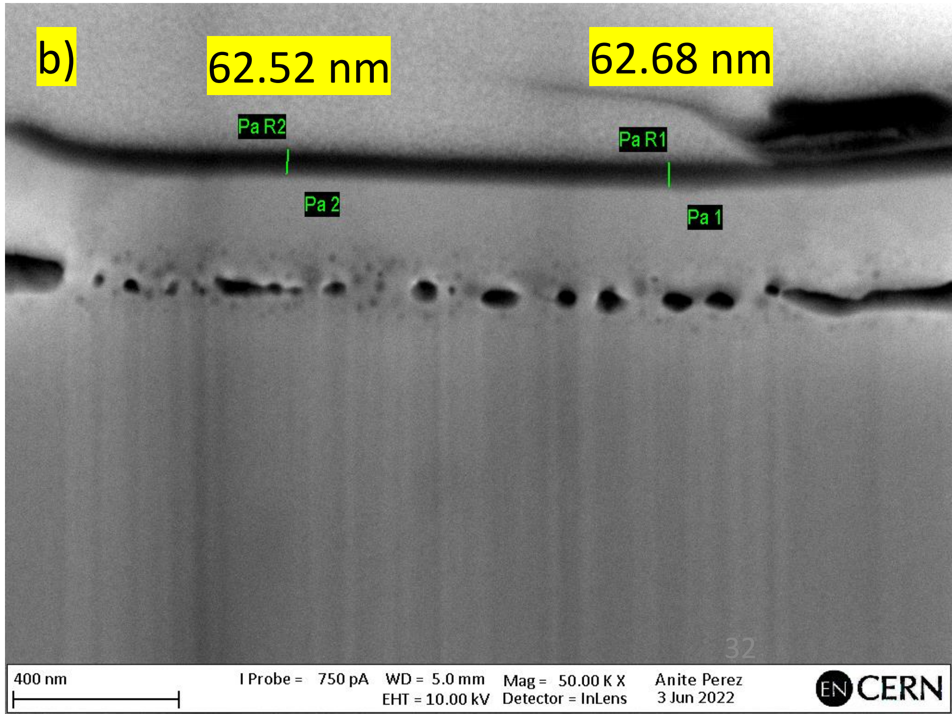
Carbon layer











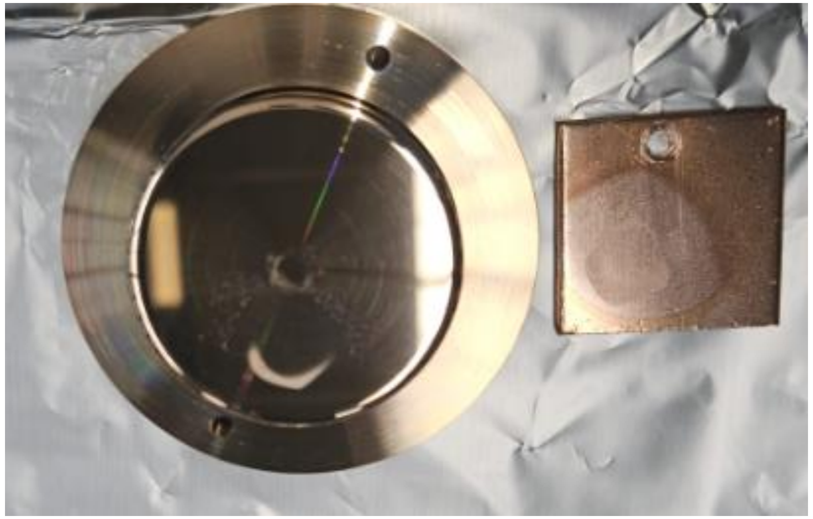
Data from a Cu sample previously analyzed after being exposed to irradiation (same parameters as the electrodes)

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

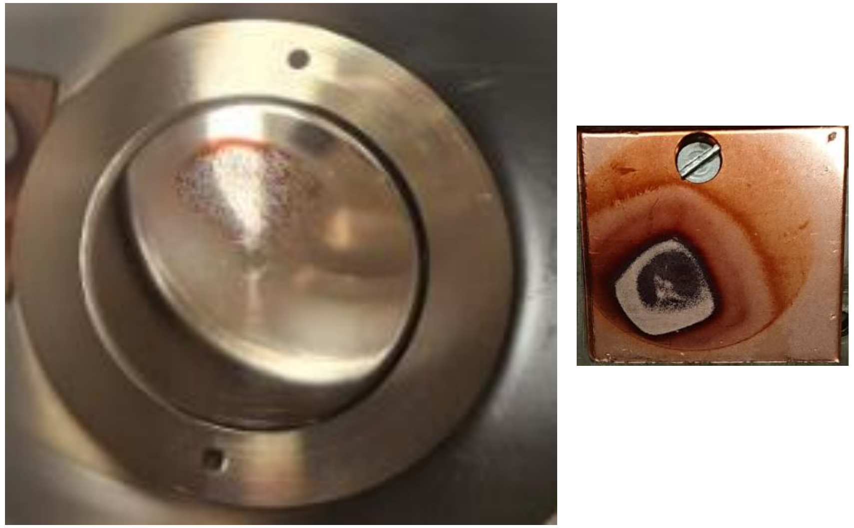
Cu layer aprox. 56 nm

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

After O_2 plasma cleaning

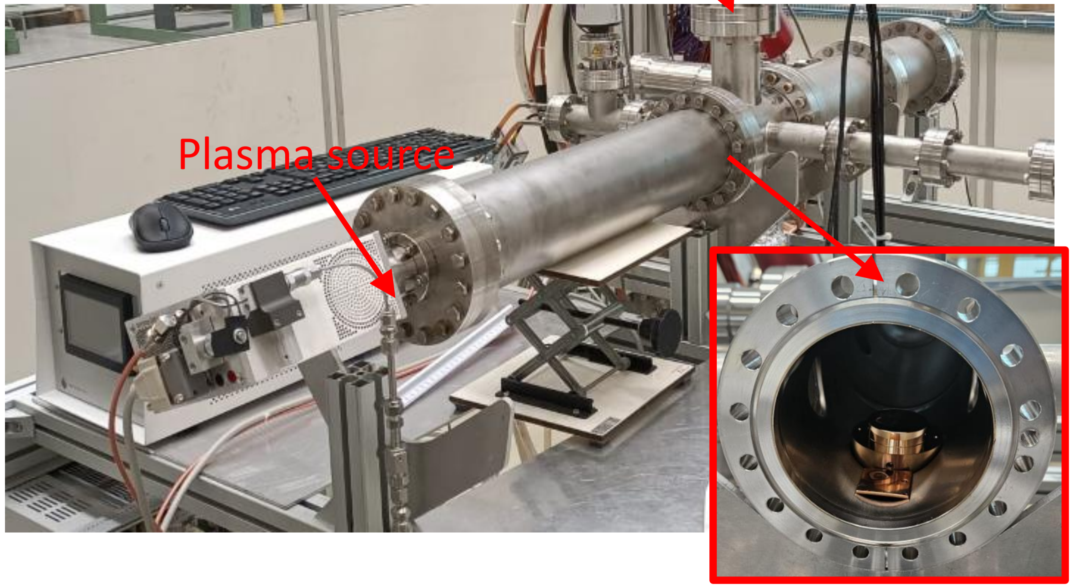


Before O_2 plasma cleaning



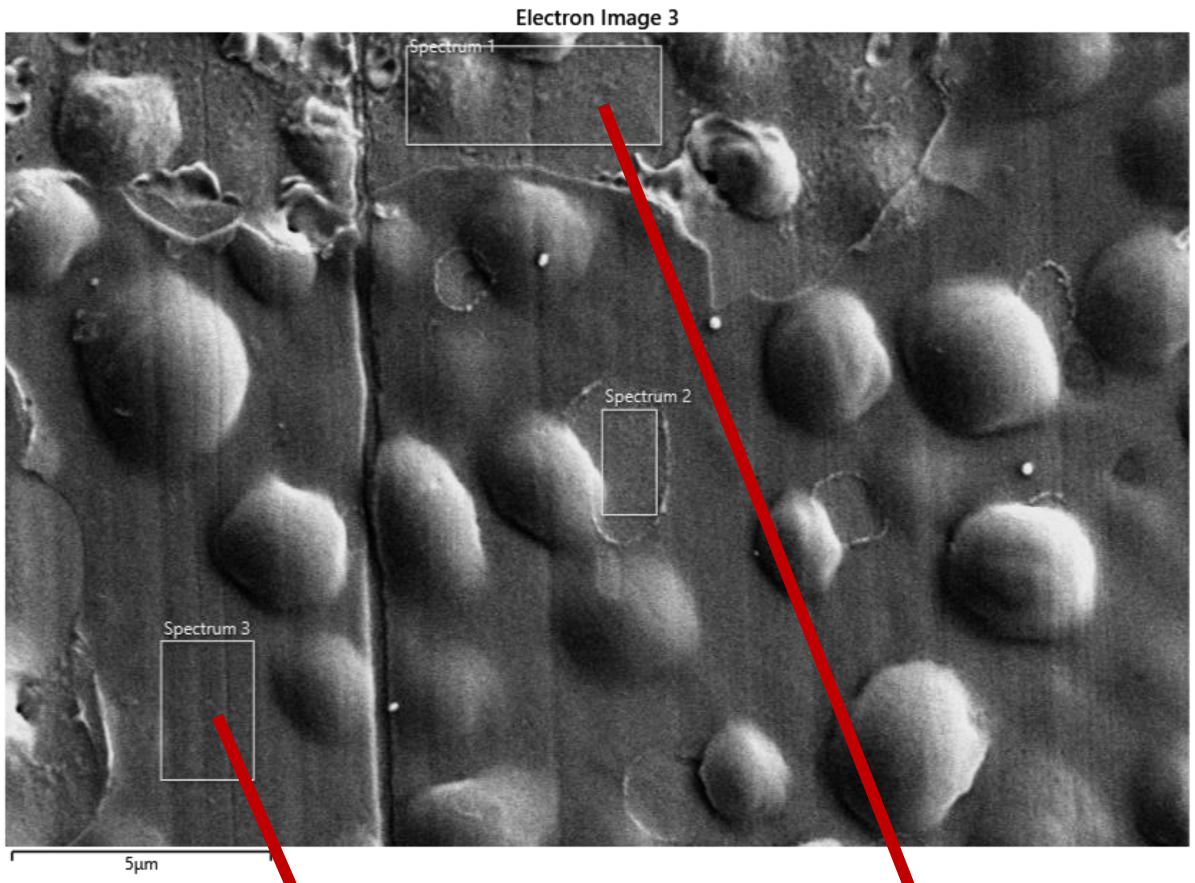
System in 181

viewport



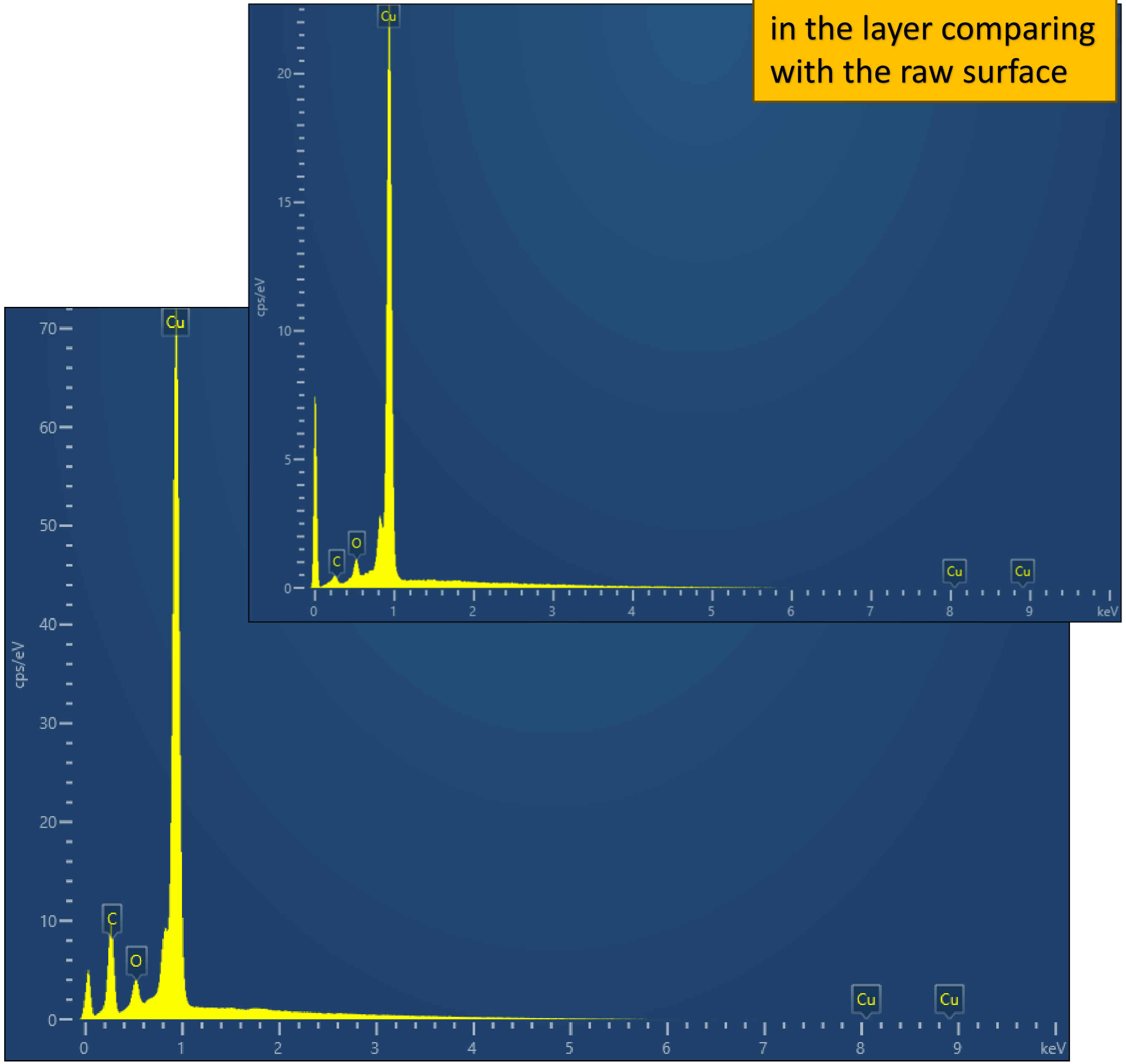
Plasma treatment parameters

Plasma source: ibss alumina tube 300W prototype, φ = 0.147 in
p = 4E-3 mbar, pure oxygen (pressure)
P_{rf} = 50 W (pl.source rf power)
L = 610 mm ± 10 mm (distance samples - plasma source); D_{ch} = 100 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)



Element	Wt%
C	12.23
O	1.1
Cu	86.68

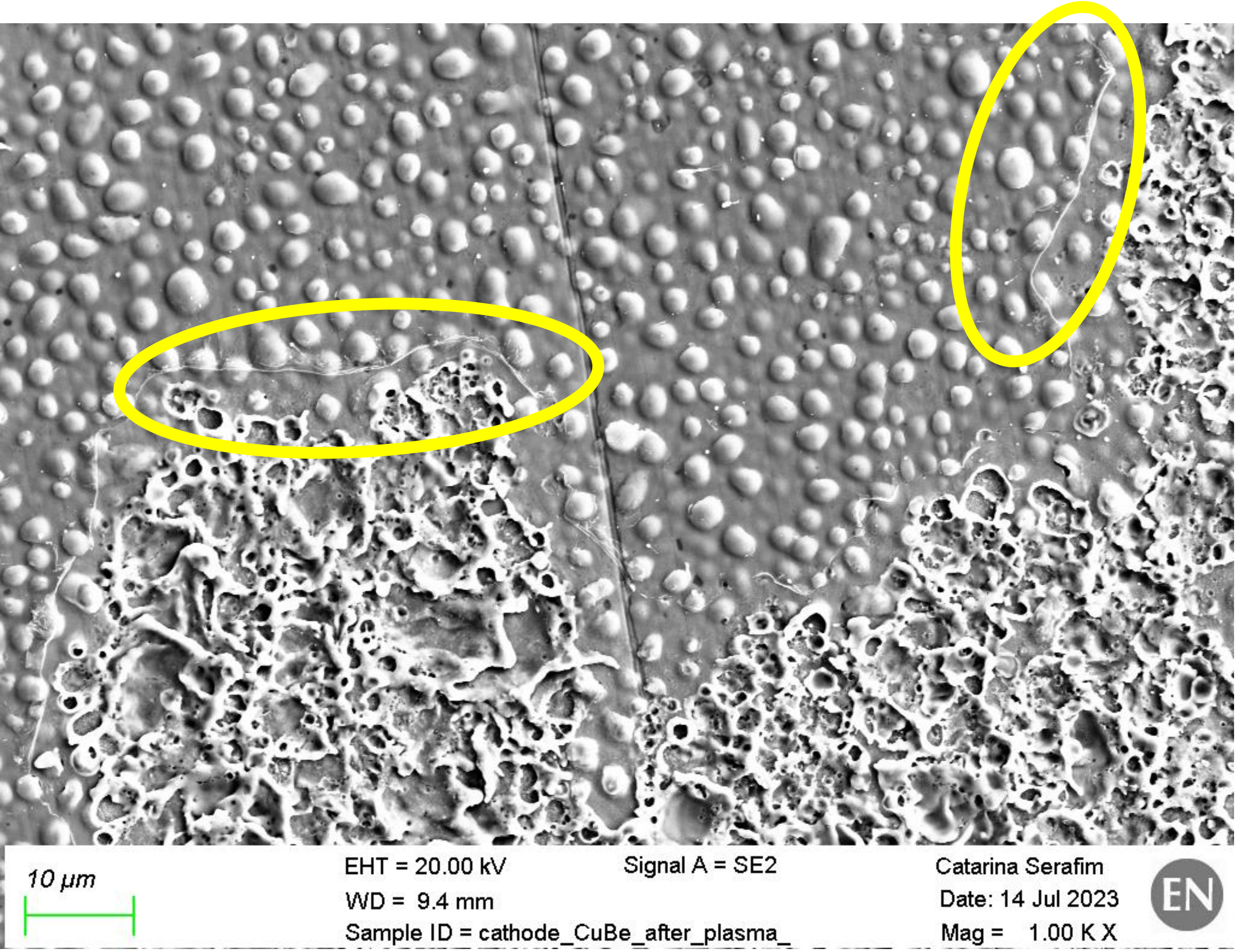
Element	Wt%
C	0.49
O	0.53
Cu	98.99

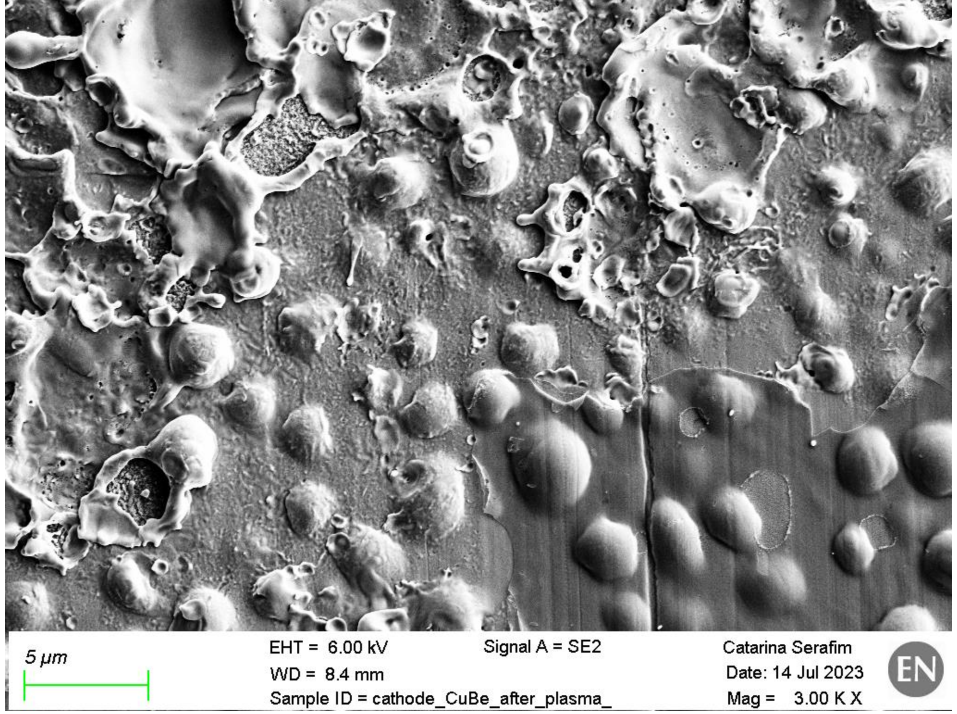


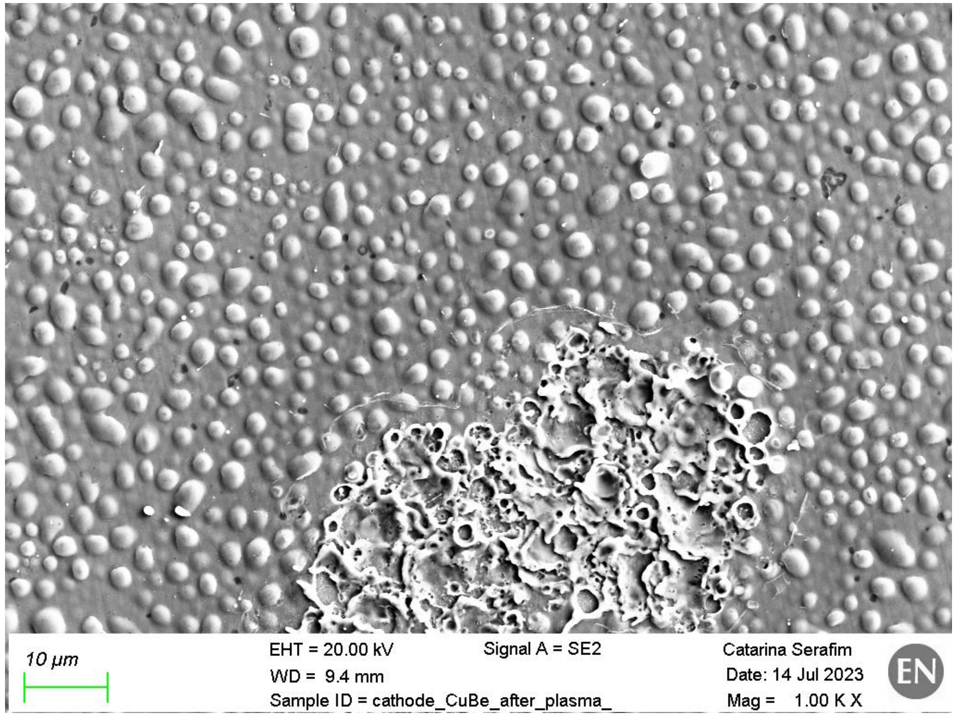
Chemical analysis

More then 12% of C in in the layer comparing with the raw surface

After plasma cleaning



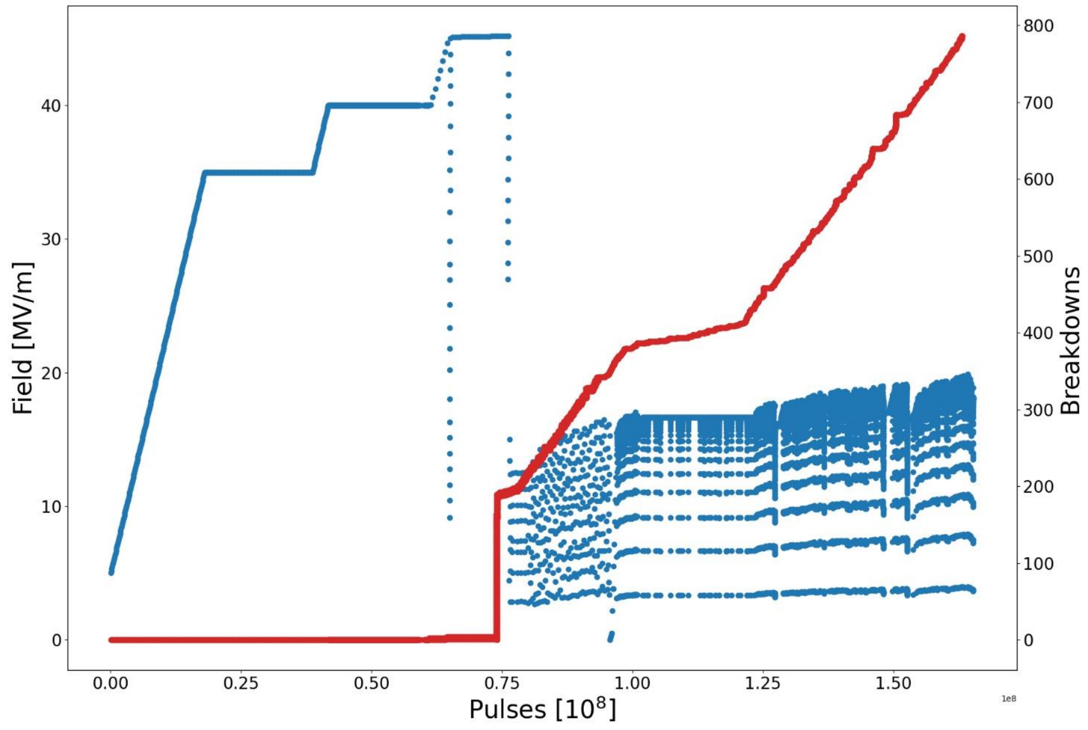


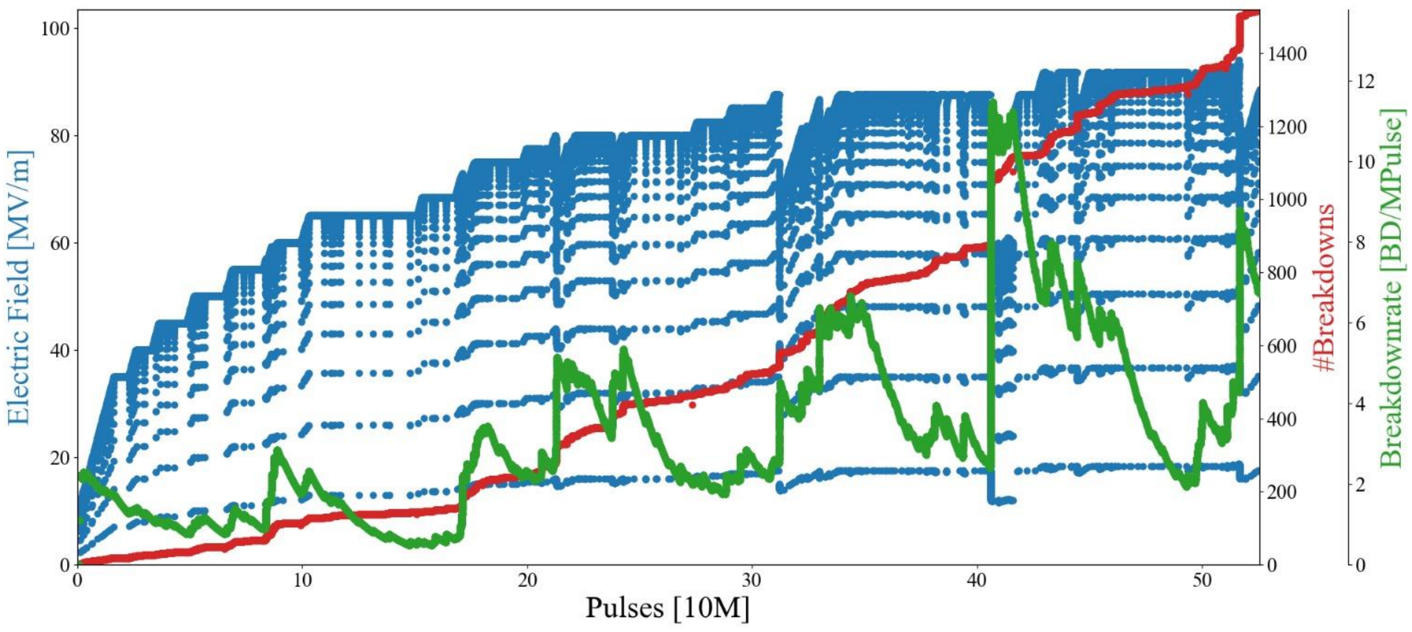


CuBe2 Breakdown Test

The followings plots are from the same set of electrodes.

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





General Conclusions of the study

Material	Max E-Field (MV/m)	Pair of electrodes	Blistering from Irradiation
Cu OFE	80	Non-irradiated pair, 2023	x
	25	Irradiated pair	Yes
TiAl6V4	110	Non-Irradiated pair	x
	95	Irradiated pair	No
CuBe2	110	Non-irradiated pair	x
	45	Irradiated pair	Yes
SS316	120	Non-irradiated pair	x
	65	Irradiated pair	No
CuCrZr	85	Non-Irradiated pair	x
	29	Irradiated pair	Yes
Nb	94	Non-Irradiated pair	x
	42	Irradiated pair	No
Ta	60	Non-Irradiated pair	x
	38	Irradiated pair	No

Results of conditioning test using high voltage pulsing

- ✓ For the non-irradiated electrodes breakdowns have shown to be dispersed throughout all the surface.
- ✓ 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.

General Conclusions of the study

	E-Field (MV/m)		Expected field (from testing non-irradiated electrodes)
Cu OFE	?	Irradiated, after plasma cleaning	80 MV/m
	25	Irradiated pair, without cleaning	
SS316	?	Irradiated, after plasma cleaning	120 MV/m
	65	Irradiated pair, without cleaning	
CuBe2	80	Irradiated, after plasma cleaning	90-110MV/m
	45	Irradiated pair, without cleaning	

Proposal was made to preform Plasma cleaning treatment in this electrodes.

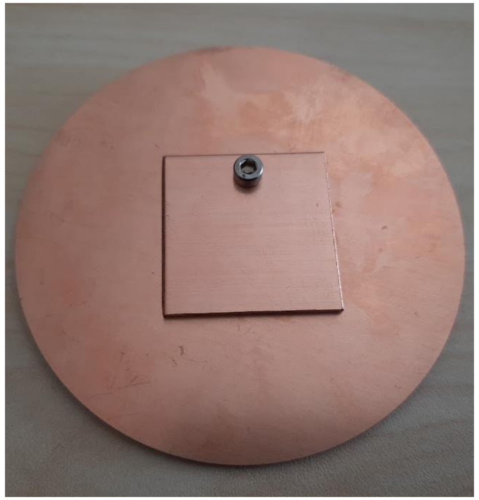
- As a pilot test, CuBe2 electrode was treated.
- After treatment, CuBe2 was tested again in the LES, where it achieved very good results.

By having good results with the pilot treatment plan, new electrodes of Cu-OFE and SS316 were submitted to irradiation and then plasma cleaned, in order to repeat the high pulsing tests. Cu-OFE is now under test and SS316 will be next.

Thank you

Installation of Cu sample for irradiation

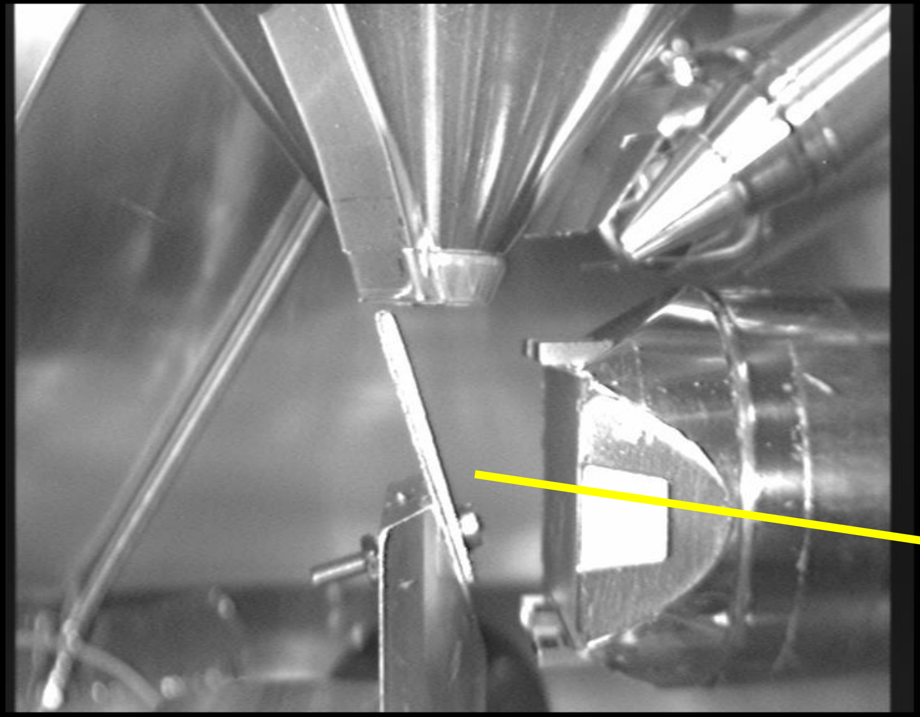
- Sample was prepared in order to be compatible with the ESBD detector.
- EBSD Mapping was preformed on the Cu sample before irradiation.

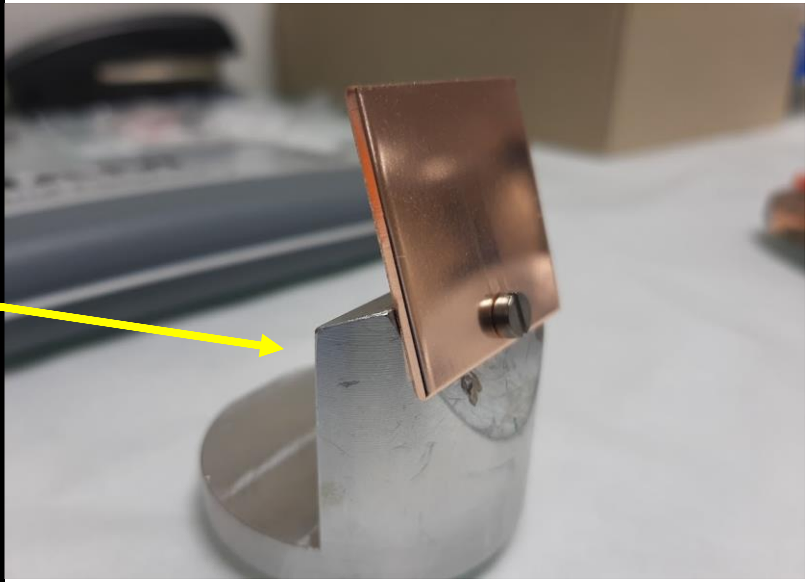


History of the sample:

- Electropolished
- Heat treated

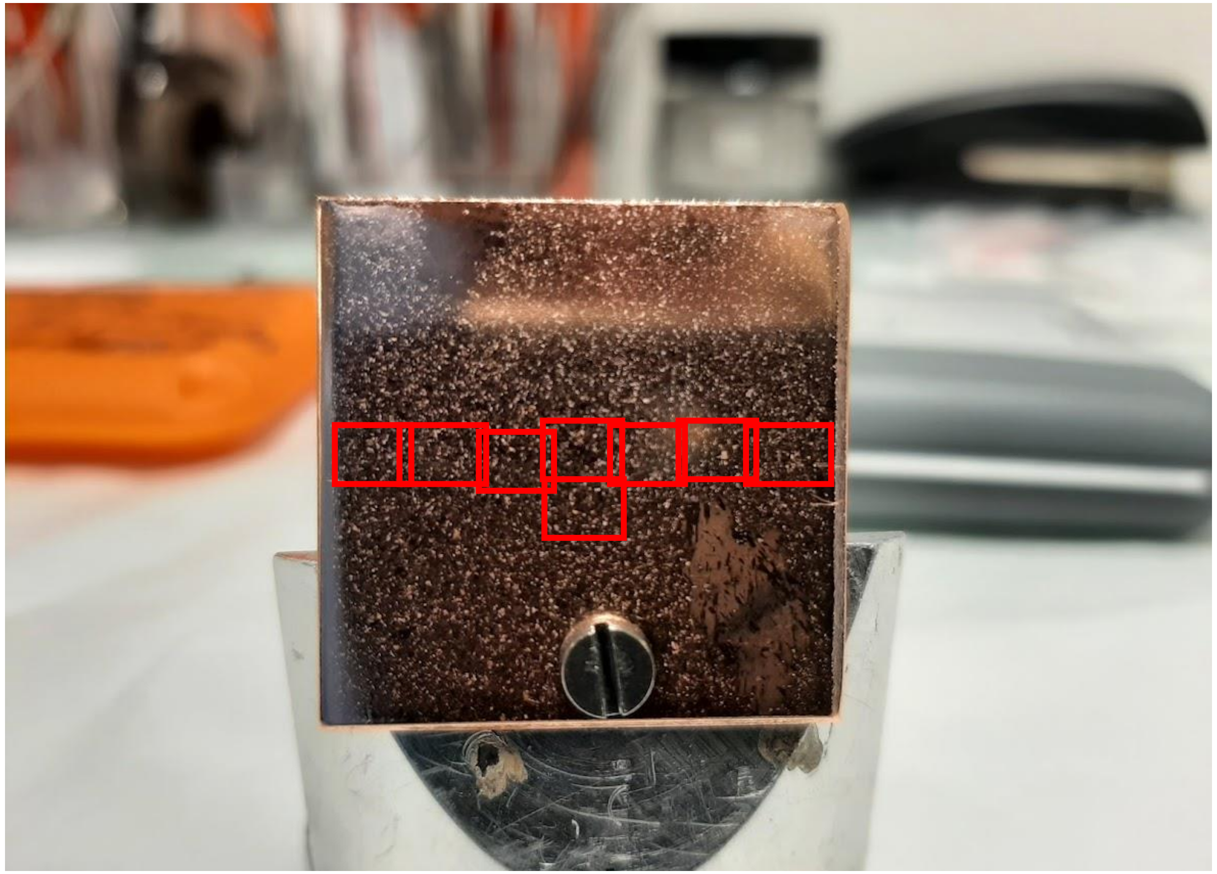
Front camera od the SEM showing the position of our sample.



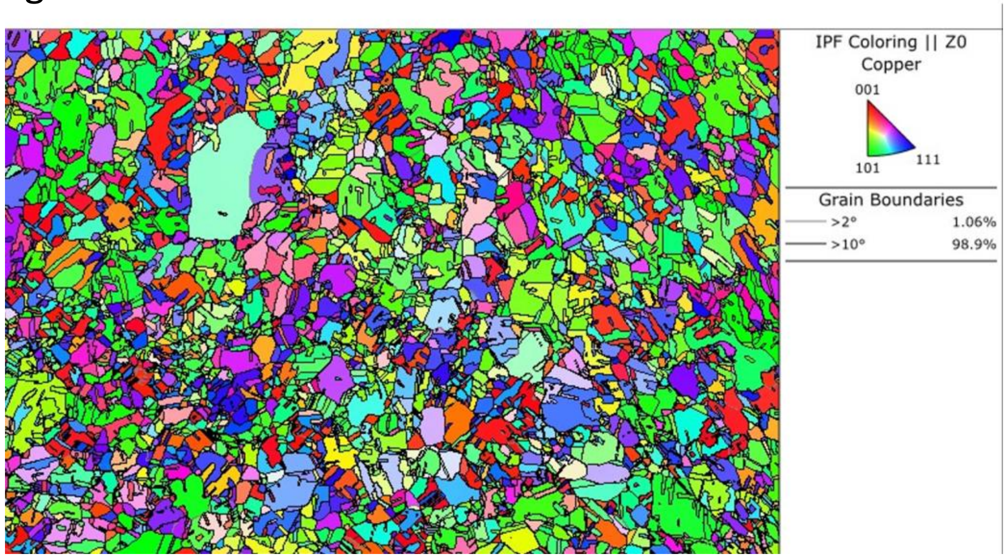


Picture from optical microscope using 80x magnification lens.

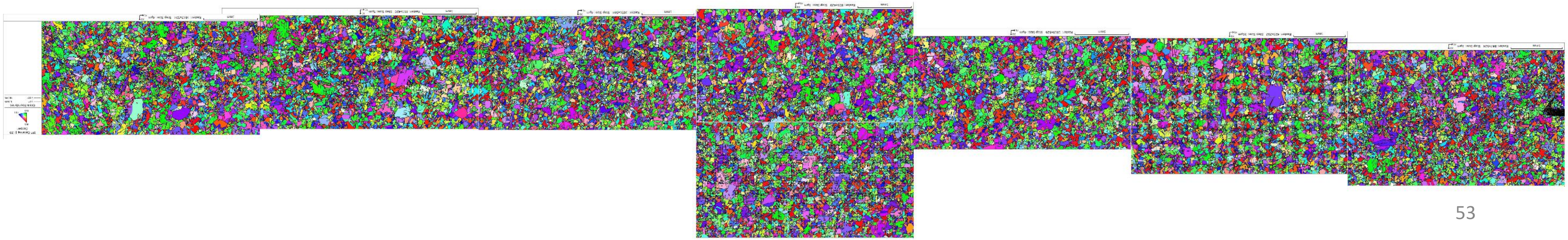
EBSD analysis



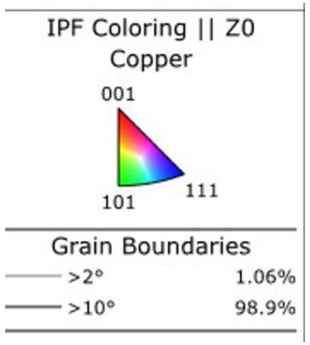
30 mm of length were covered on the analysis with different pixels step sizes between 10 and 4 microns, allowing us to have the indexation of the different grains.

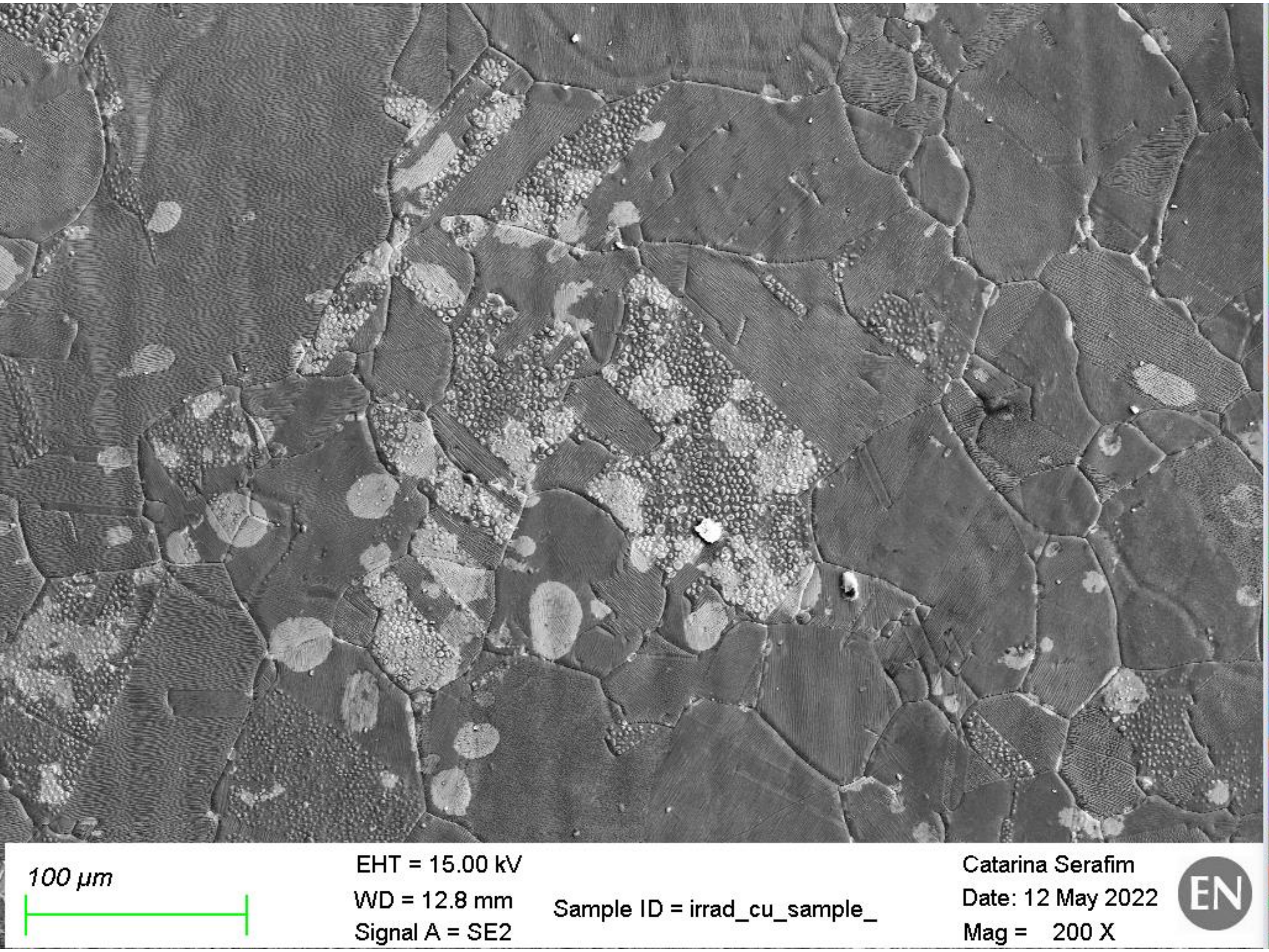


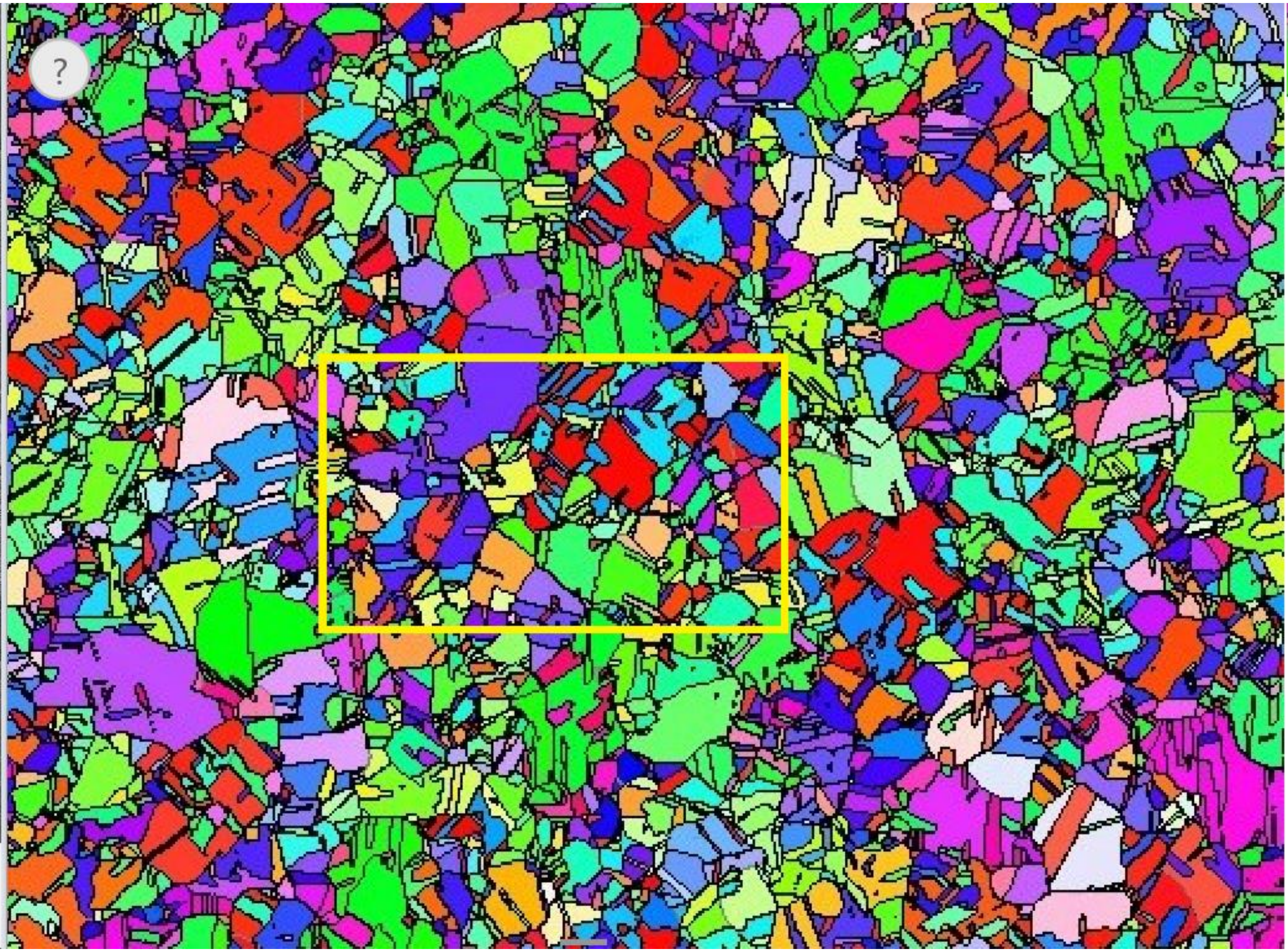
Stitching map result from the EBSD analysis.



- The grains presenting red color were associated with high density of blisters.
- Grains presenting blue and green colors have showed to have less blisters/no blisters.





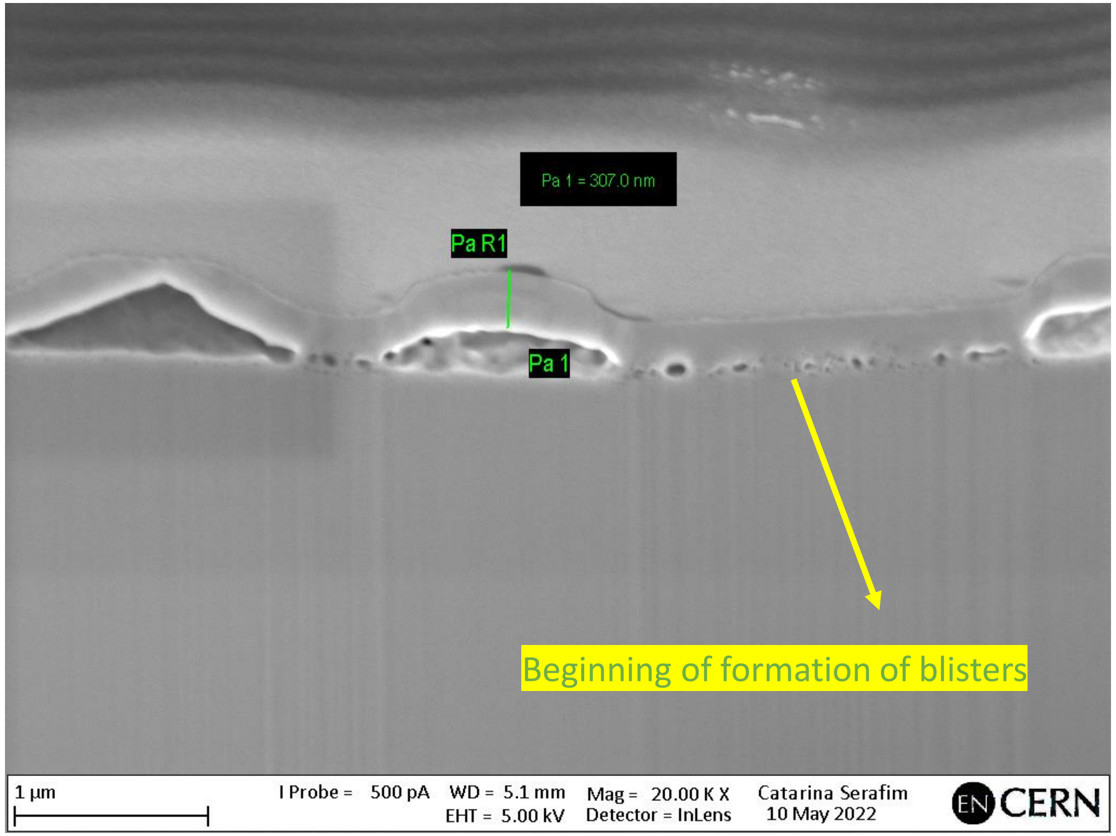


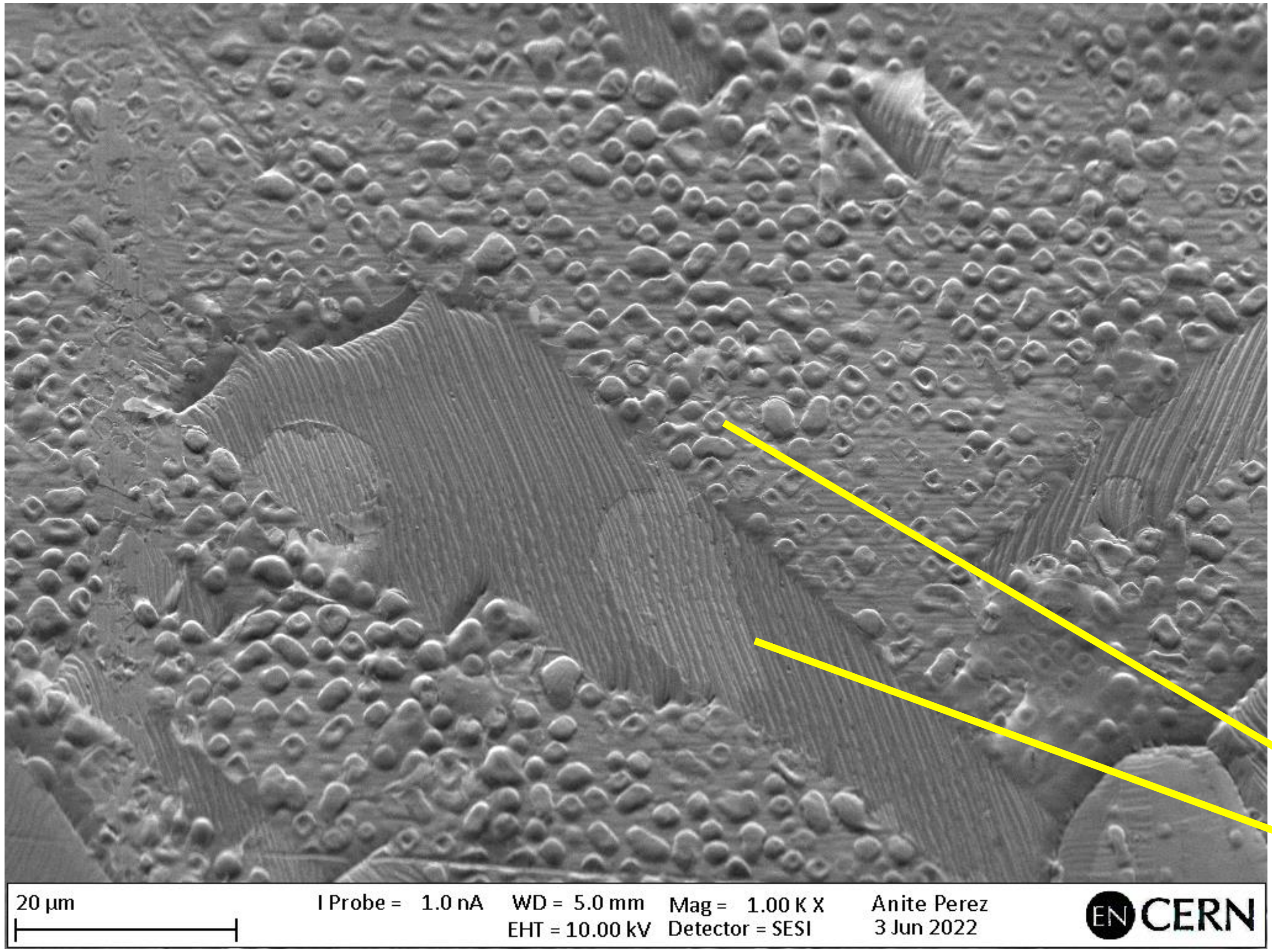
For the blistering phenomena a parallel work is under study.

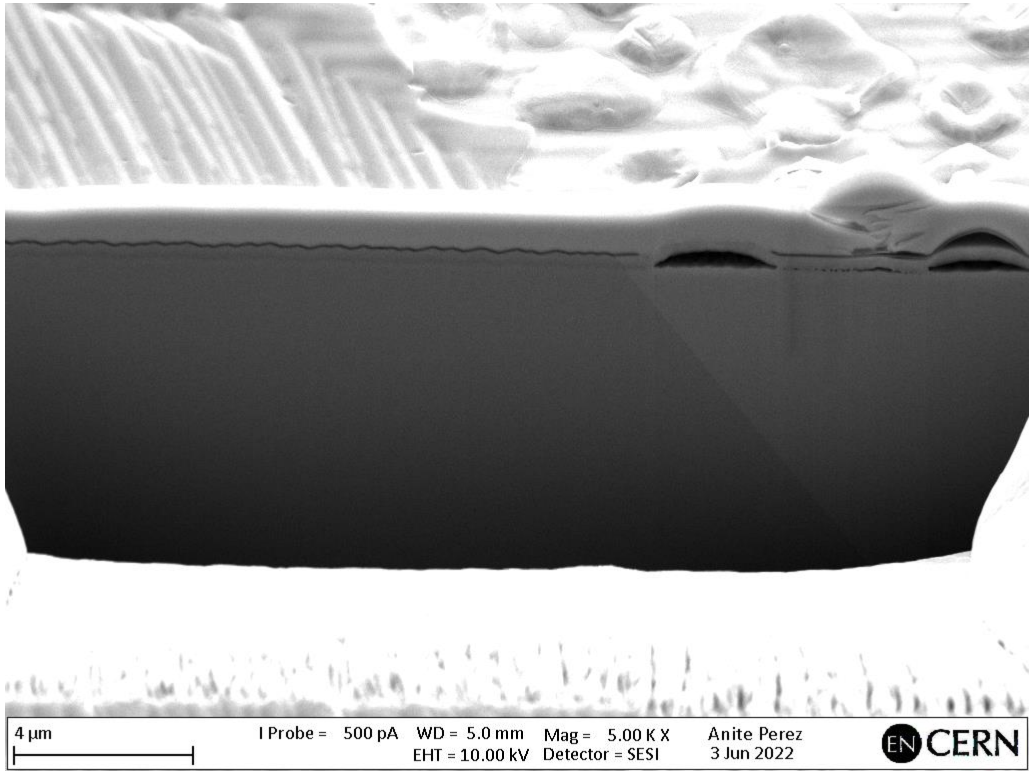
- Trying to understand correlation between **different grains orientation vs blistering**.
- Irradiated with same beam parameters Cu sample – doing analysis with EBSD mapping and FIB/SEM

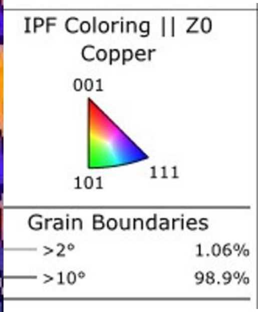
<u>We are studying 3 main orientation:</u>

- 001 orientation – where we see the high density of blisters
- 101 orientation – where we see lower density of blisters
- 111 orientation – where we see few/no blisters









Red grain = orientation 001
Blue grain = orientation 111