Recent microscopy at CERN

Enrique Rodríguez Castro

Microscopy at CERN	**Introduction**	**Equipment**	OM	
			SEM	
			FIB-SEM	
Microscopy for CLIC	**Quality control**	**Machining quality**		
		Pollution		
	Studies	**LES**		
		Dislocation	Reference	
			Fatigued	
			LES electrode	
		Post-Mortem	LES	Reference
				1 BD
				2 BD
			T24 Open	Brazing
				Iris
				Mechanical marks

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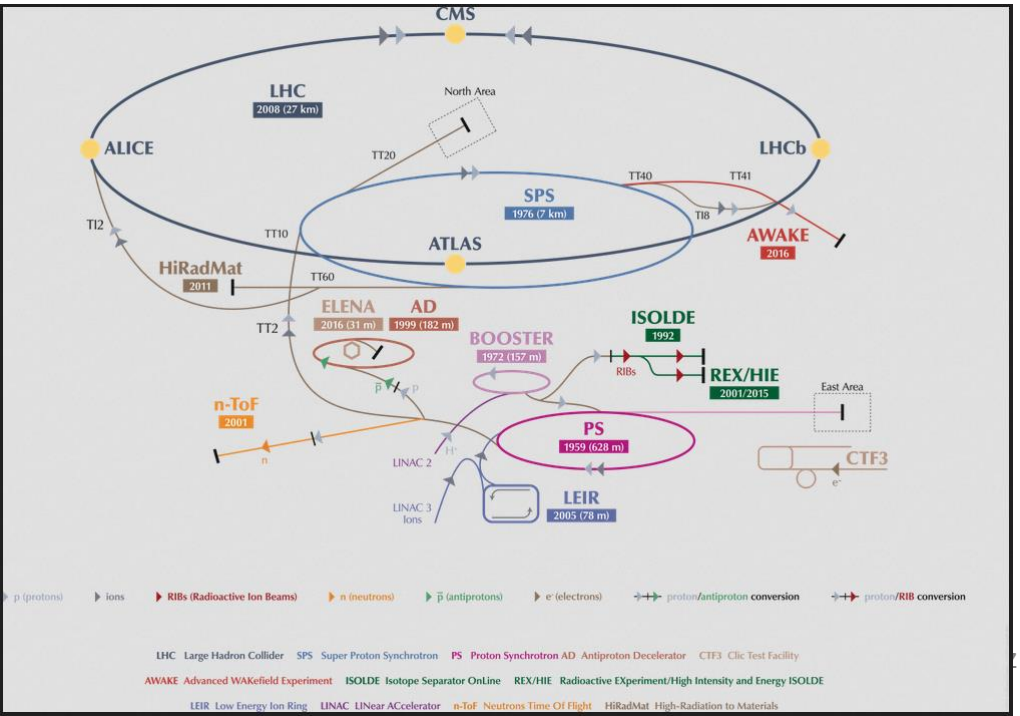
Microscopy at CERN

5 sectors ⊃ Accelerator & Technology (A&T)

↳ 4 departments ⊃ Engineering (EN)

↳ 9 groups ⊃ Mechanical & Materials Engineering (MME)

↳ 6 sections ⊃ Metallurgy, Metrology (MM) & NDT



Experiments and projects

- CERN experiments (Grey Book)
- ACE
- AEGIS
- ALICE
- ALPHA
- ASACUSA
- ATLAS
- ATRAP
- AWAKE
- CAST
- CERN Neutrino Platform
- CLIC
- CLICdp
- CLOUD
- CNGS
- CMS
- COMPASS
- DIRAC
- ELENA
- FCC
- GBAR
- HL-LHC
- HiRadMat
- ISOLDE
- LHCb
- LHCf
- LIU project
- MoEDAL
- NA61/SHINE
- NA62
- NA64
- nTOF
- PBC
- SHiP
- TOTEM
- UA9
- WLCG

Optical Microscopes

StereoMicroscope: M205C by LEICA

Objectives	
Resolution	Max. 1,050 lp/mm
Magnification	7.8×–1,280×
Object field	Max Ø 59mm
Field Optics Illumination	Bright Field (BF), Dark Field (DF), Single-Side Dark Field with Rotterdam contrast and Constant Color Intensity Control (CCIC)

Digital microscope: VHX 1000E by KEYENCE

Features	
Resolution	2-6 million pixels: 1600 (H) x 1200 (V) ≈ 1000 TV lines 8 million pixels: 3200 (H) x 2400 (V) ≈ 1600 TV lines 18-54 million pixels: 4800 (H) x 3600 (V) ≈ 2000 TV lines
High dynamic Range (HDR)	16-bit resolution through RGB data from each pixel
Magnification	1-1000x
Field Optics Illumination	*DF, *BF, *PL and *DIC

Optical Microscope: AxioImager by ZEISS

Performances	
Resolution	At 1296 X 968 Resolution of 3 Frames/S At 430 X 322 Resolution of 11 Frames/S At 258 X 193 Resolution of 16 Frames/S
Magnification	12.5 – 1,500x
Field Optics	Dark Field (DF), Bright Field (BF), Polarized light (PL), Differential Interference-Contrast (DIC)

Software
AxioVision & Zencore (New version)

Electron microscopy

Scanning Electron Microscope (SEM) ΣIGMA by ZEISS

Performance	
Resolution	1.2 nm at 30kV
Acceleration Voltage	0.1 – 30kV
Magnification	12 – 500,000x

Specimen, Chamber and Stage	
Stage Movement range	5 motorized axes: X,Y,Z,T and R X and Y: ≥ 125 mm; Z: ≥ 50 mm T: 0° to 90°; R: 360°
Maximum specimen Weight	Up to 0.5 kg (tilted) and 2.0 kg (not tilted)
Chamber Internal Dimensions	365 mm diameter x 275 mm high

Focused Ion Beam (FIB) XB540 by ZEISS

SEM Performance	
Column	Crossbeam 540 (Gemini® II column)
Resolution	High resolution configuration: 0.7 nm at 30 kV
Acceleration Voltage	0.02 - 30 kV
Magnification	12x - 2,000,000x

FIB Performance	
Source	Gallium Ion
Resolution	3nm at 30kV
Acceleration Voltage	0.5 – 30 kV
Magnification	300x - 500,000x

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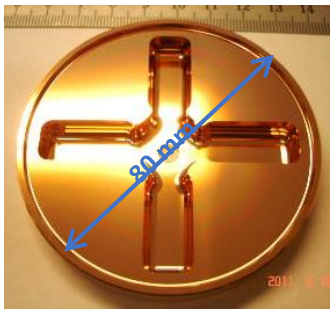
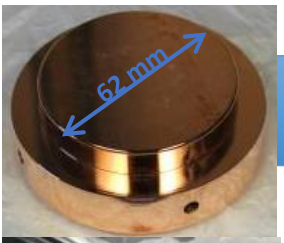
Microscopy for CLIC



UP-Diamond machining

Cleaning
Pollution analysis

SEM
(QC of the surface)

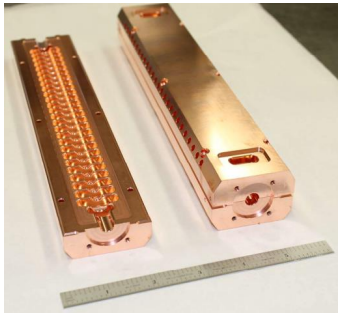


LES
HARD-Cu

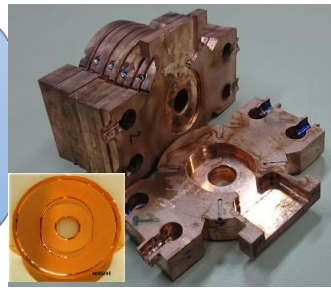
Assembly:
H_2 diffusion bonding/brazing

LES
SOFT-Cu

Vacuum baking

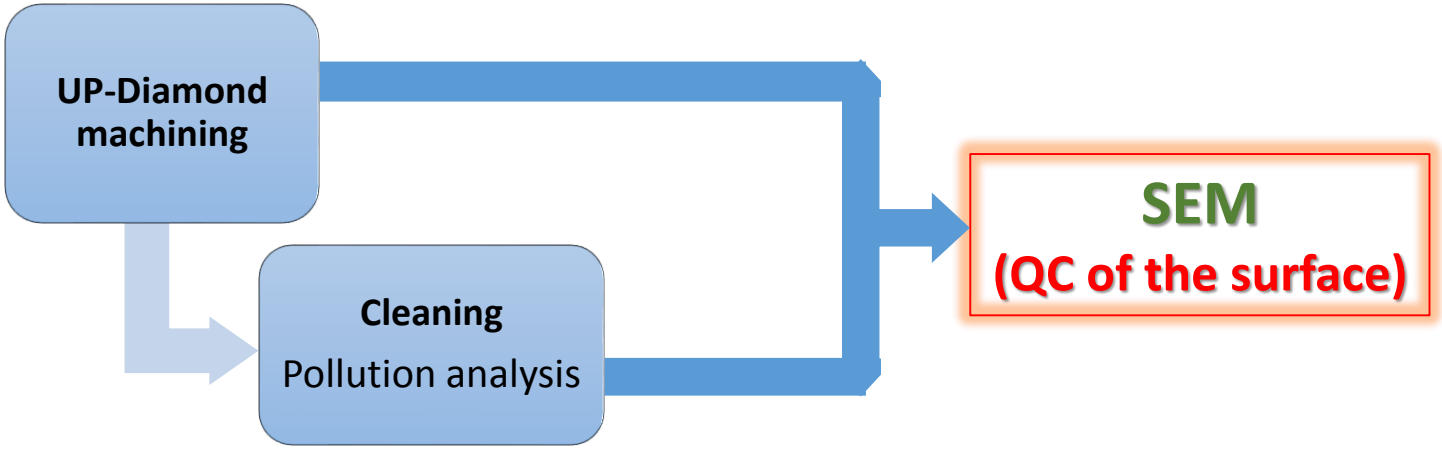


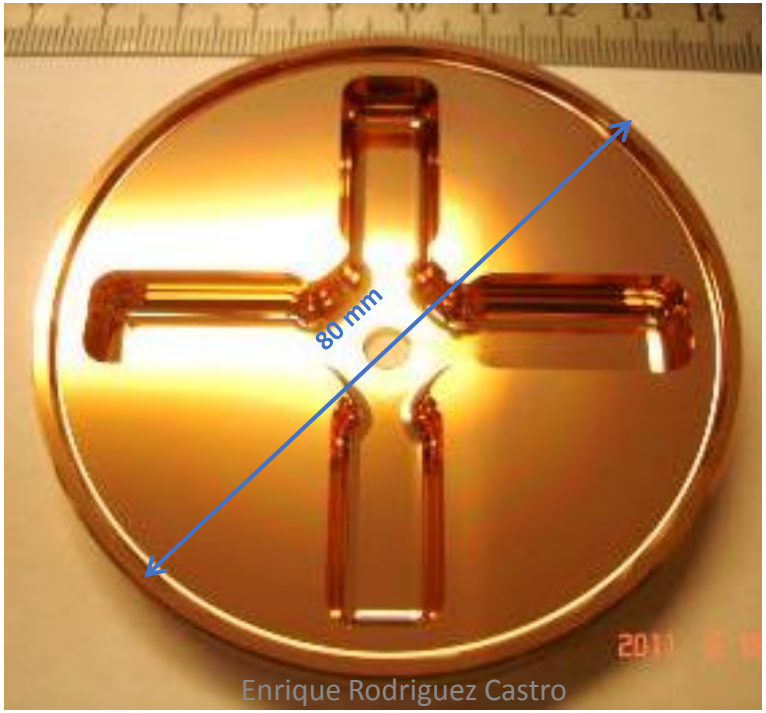
High Gradient testing



SEM
(Post-mortem)

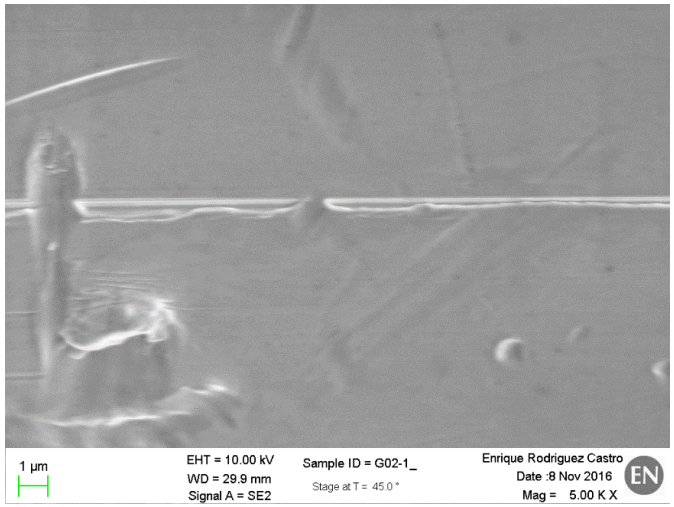
Microscopy for CLIC

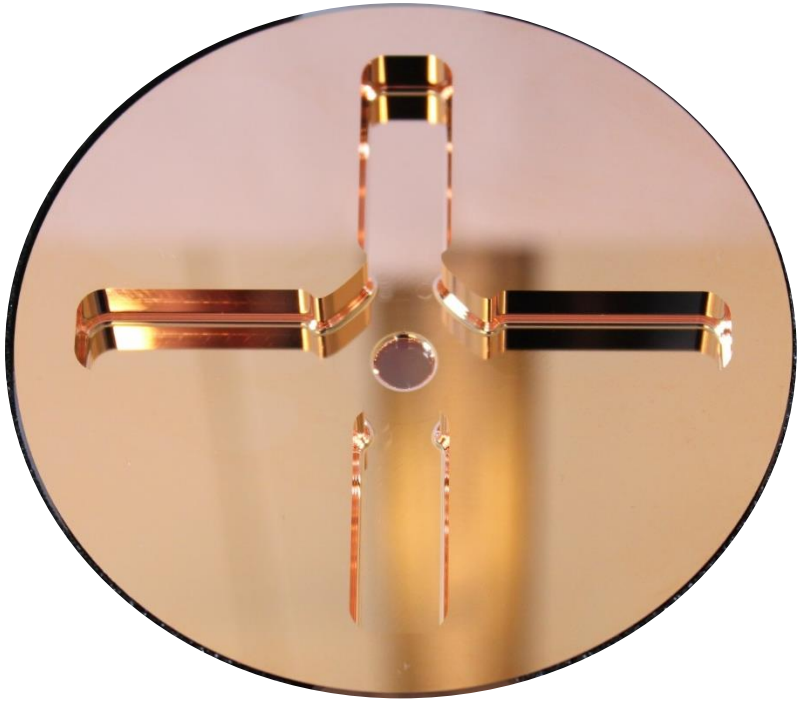




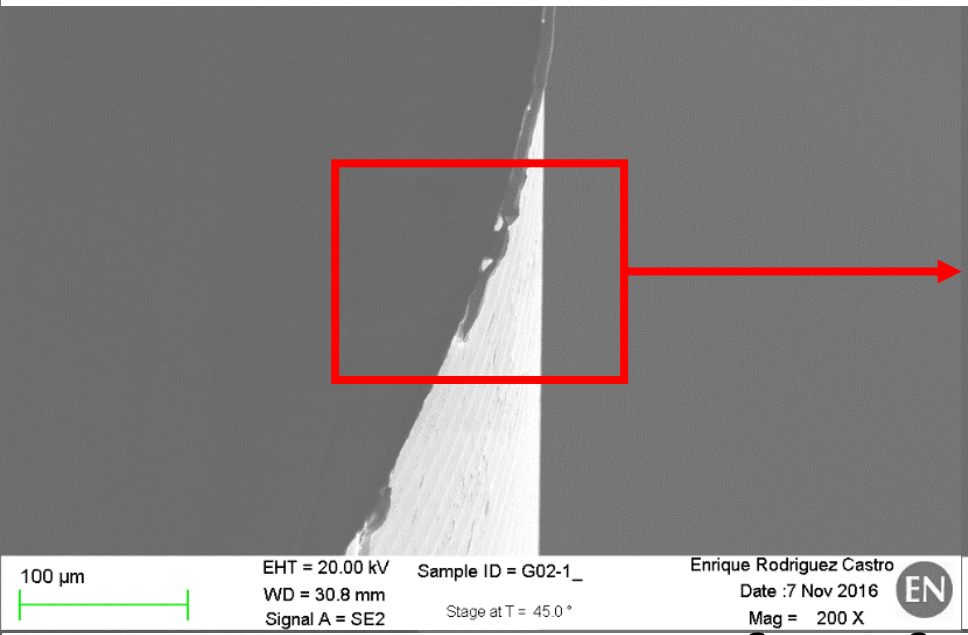
Machining qualification

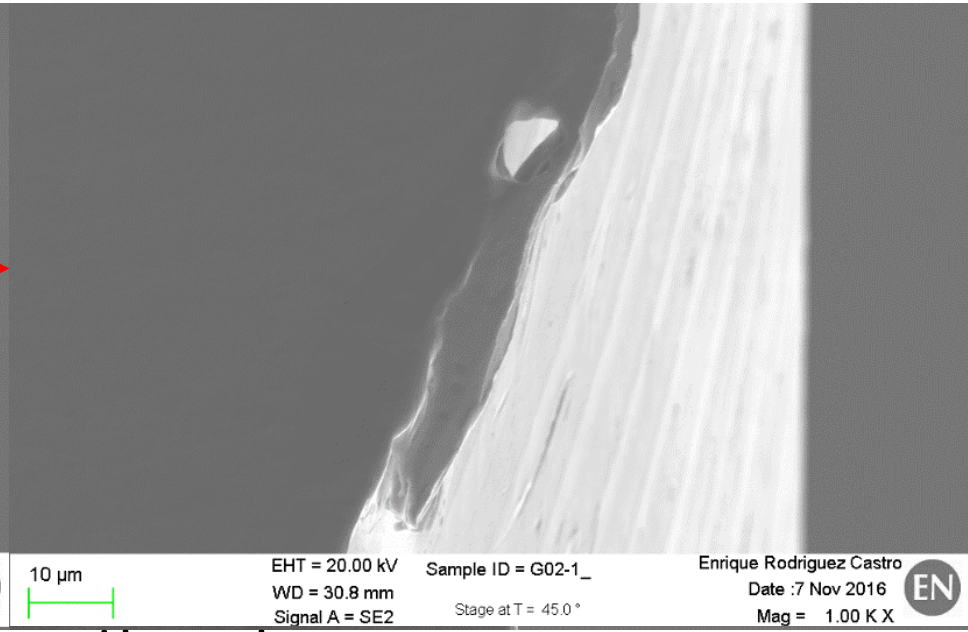
- Important to feedback company
  - Improvement on machining process
  - Correction of errors (before finishing the manufacturing)



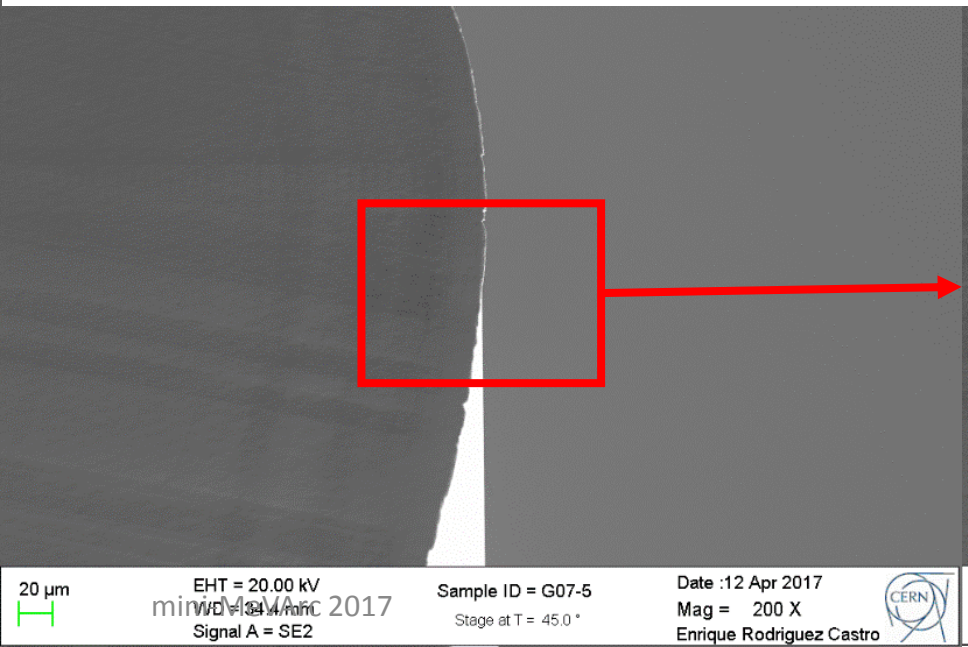


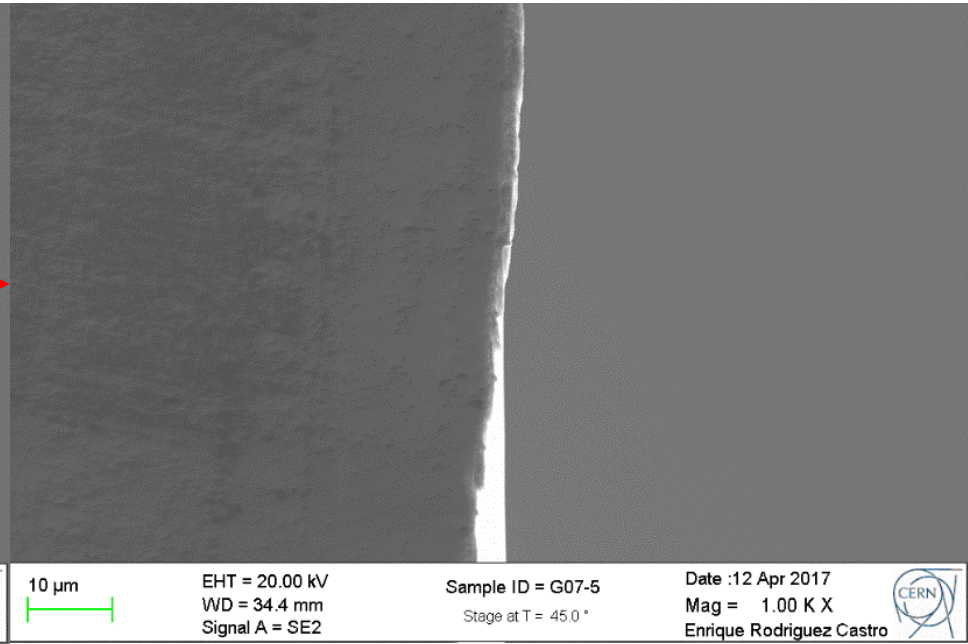
First observation





After feedback





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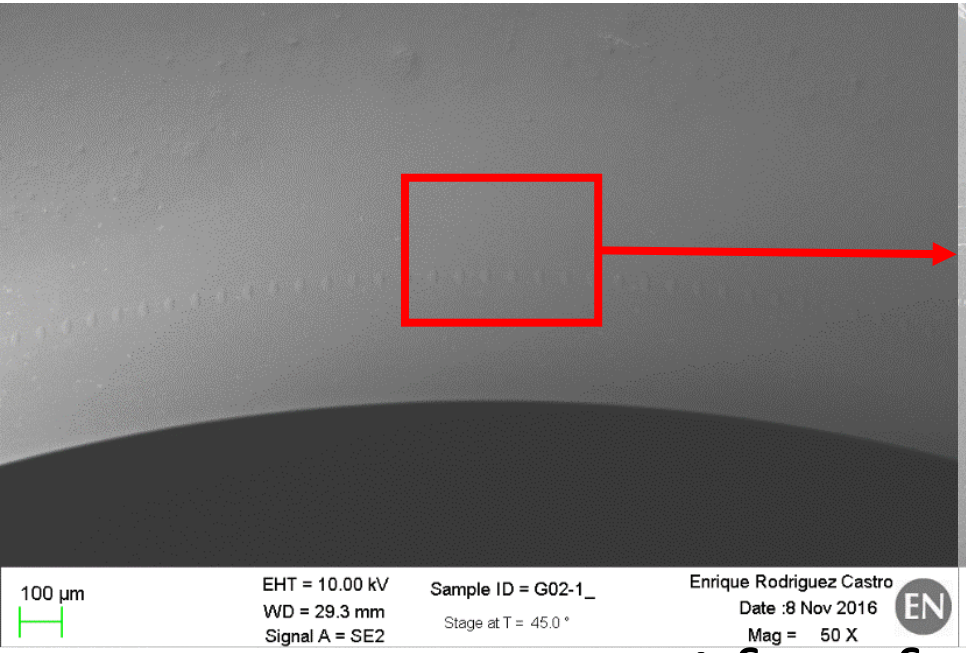
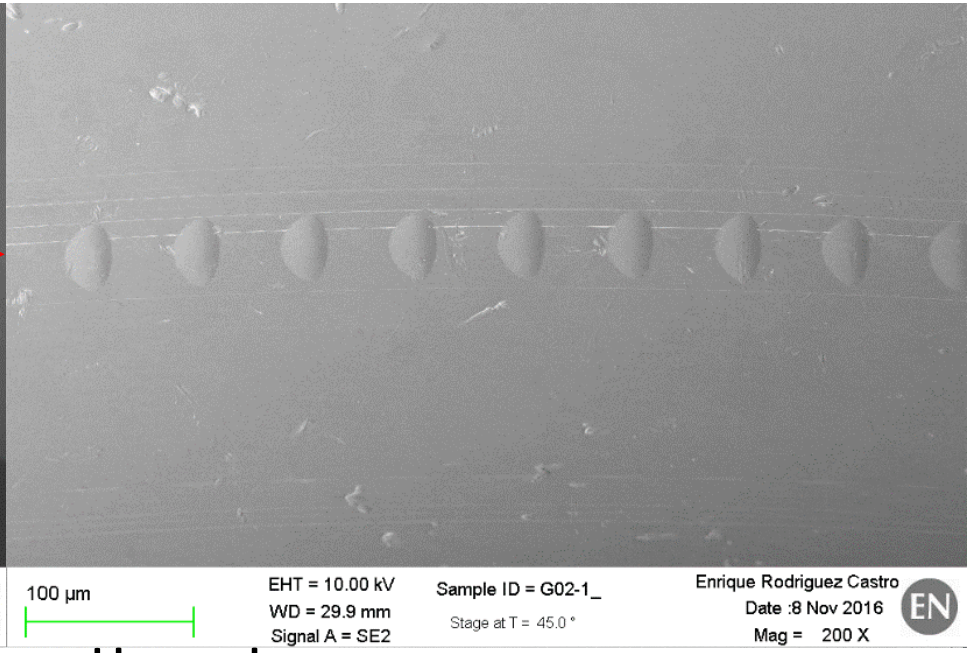
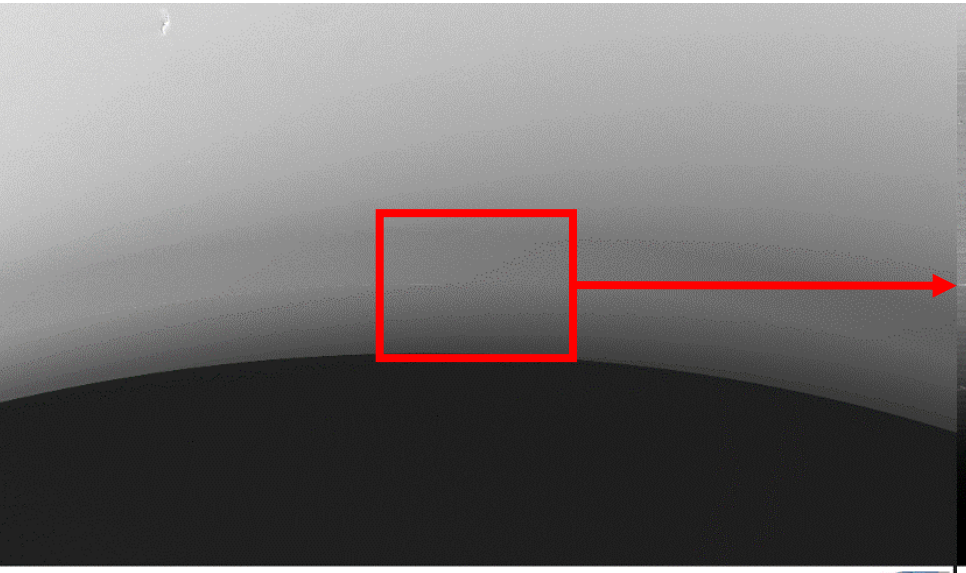
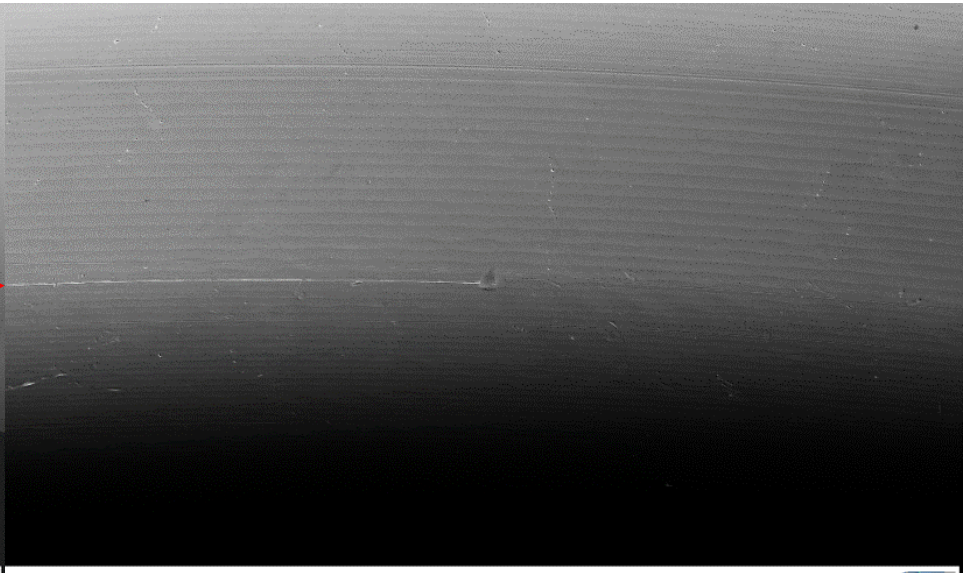




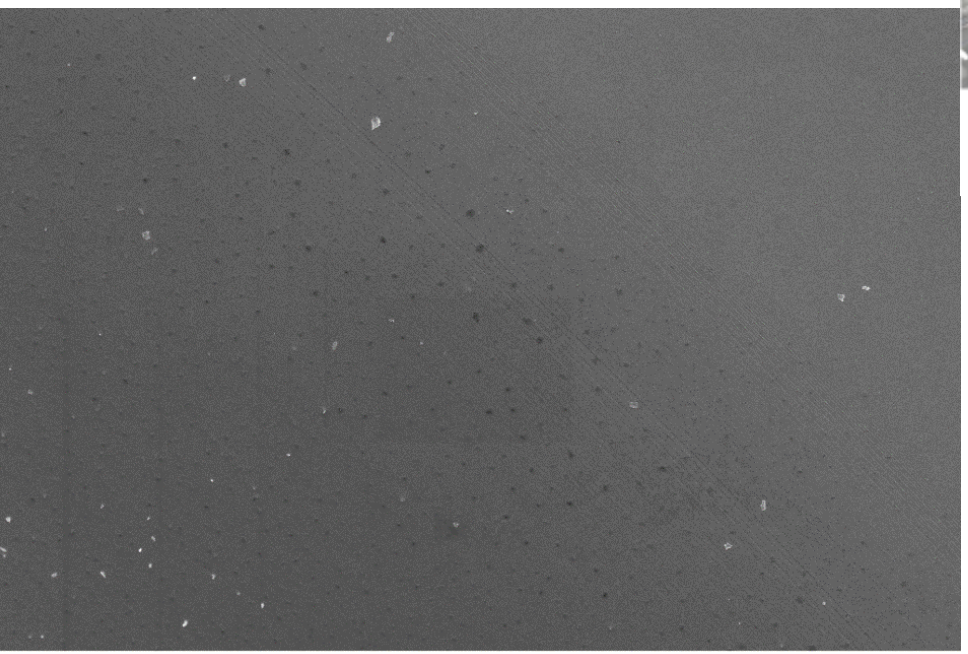
After feedback

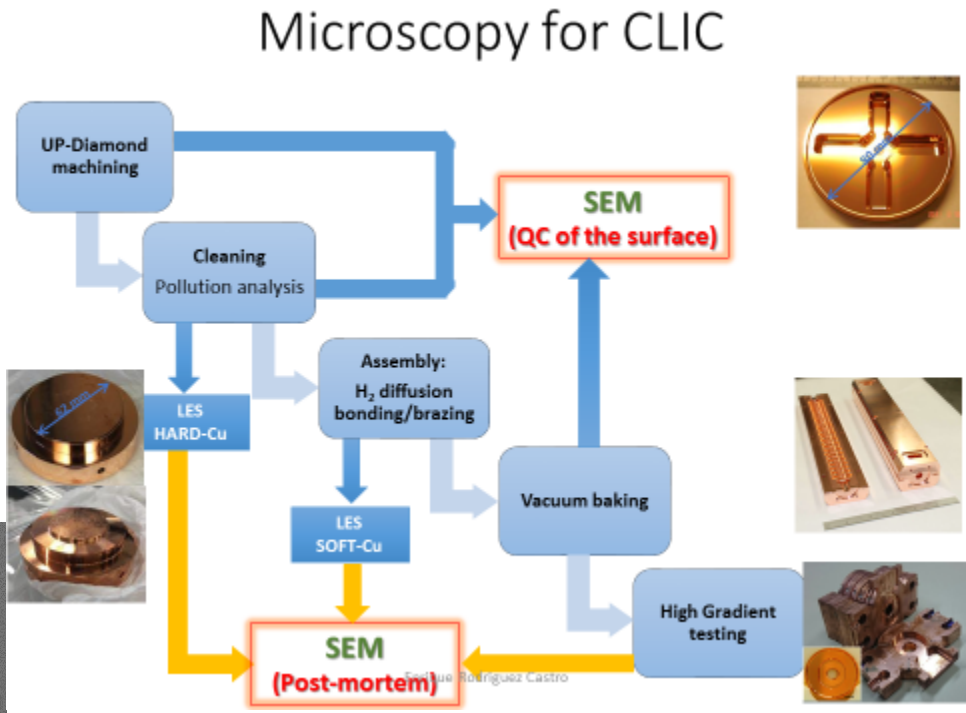




Pollution Analysis

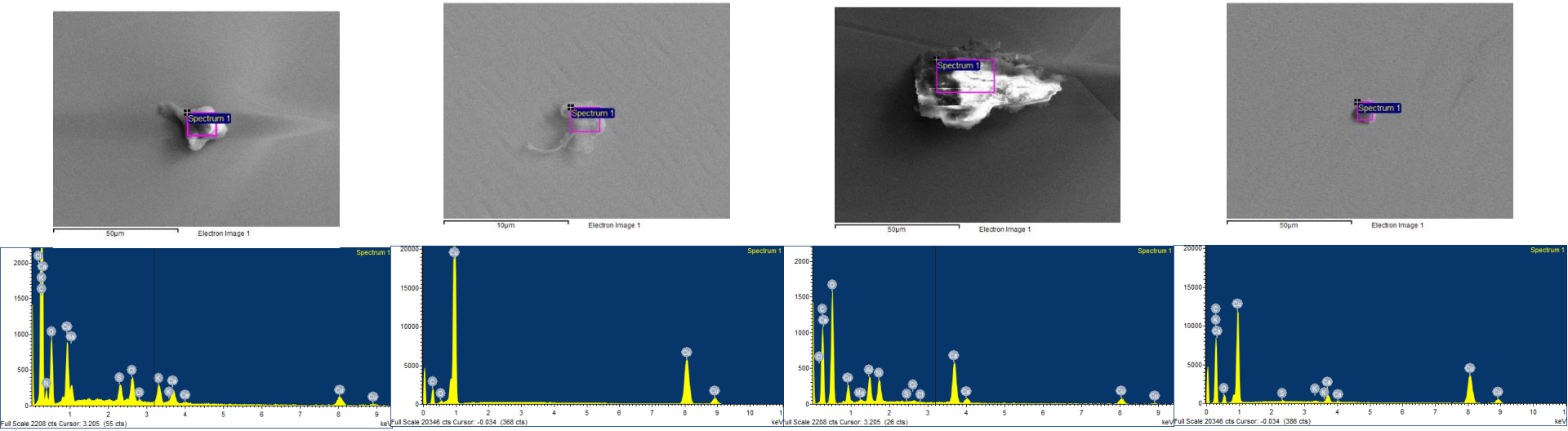
- A particle on surface could be the origin of a BD.
- Reduce the contamination on structures is crucial.
- Procedure for furnace qualification (including CERN new oven).





- Possibility to trace back if any problem is encountered

Current situation



- Tedious and slow process → one SOI at a time
- Impossible to scan all surface → qualitative

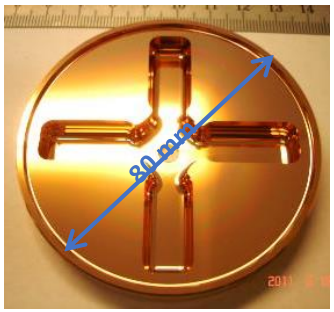
APA (Automated Particle Analysis)

- Huge increment of number of particles that can be analysed.
- Possibility to create custom classification → (thus ignore non important SOI or enhance those with dangerous contaminants)
- Shape recognition.

	APA	Manual
Number of analysed particles	21 290	50
Number of classified particles	20 225	/
% of fitting	95 %	/

- From qualitative to quantitative

Microscopy for CLIC



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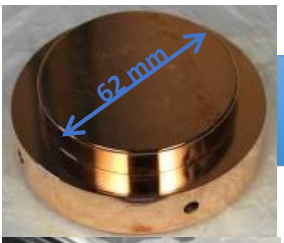

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HARD-Cu

Assembly:
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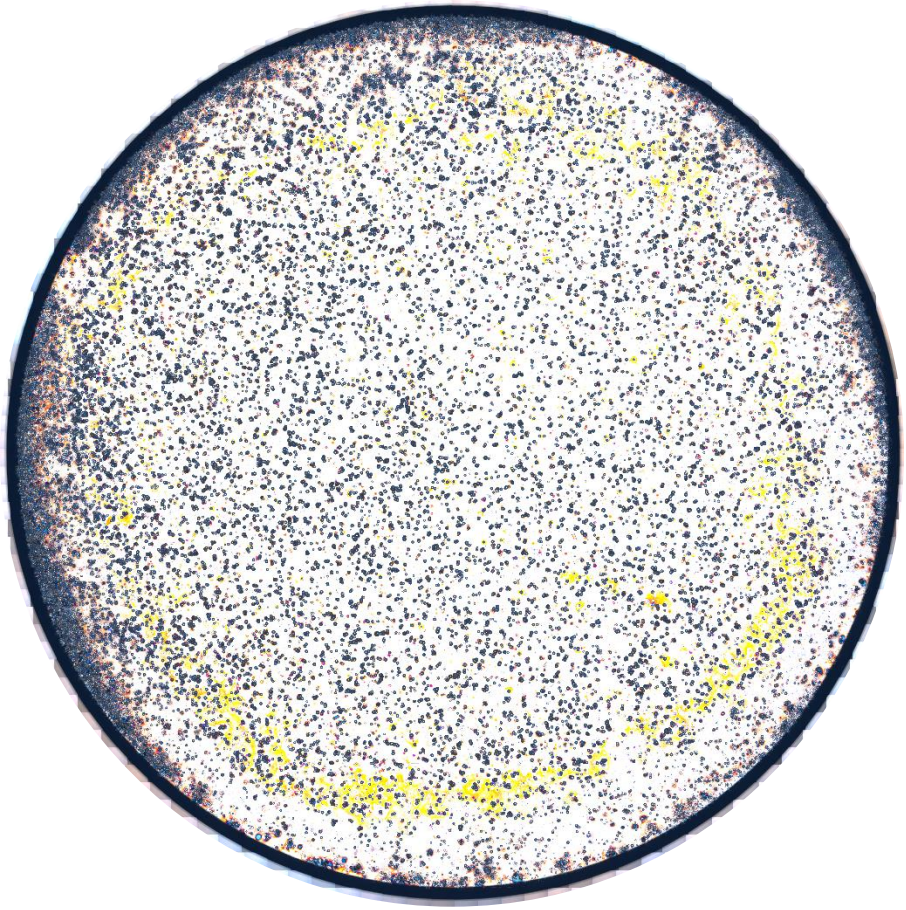
SOFT-Cu

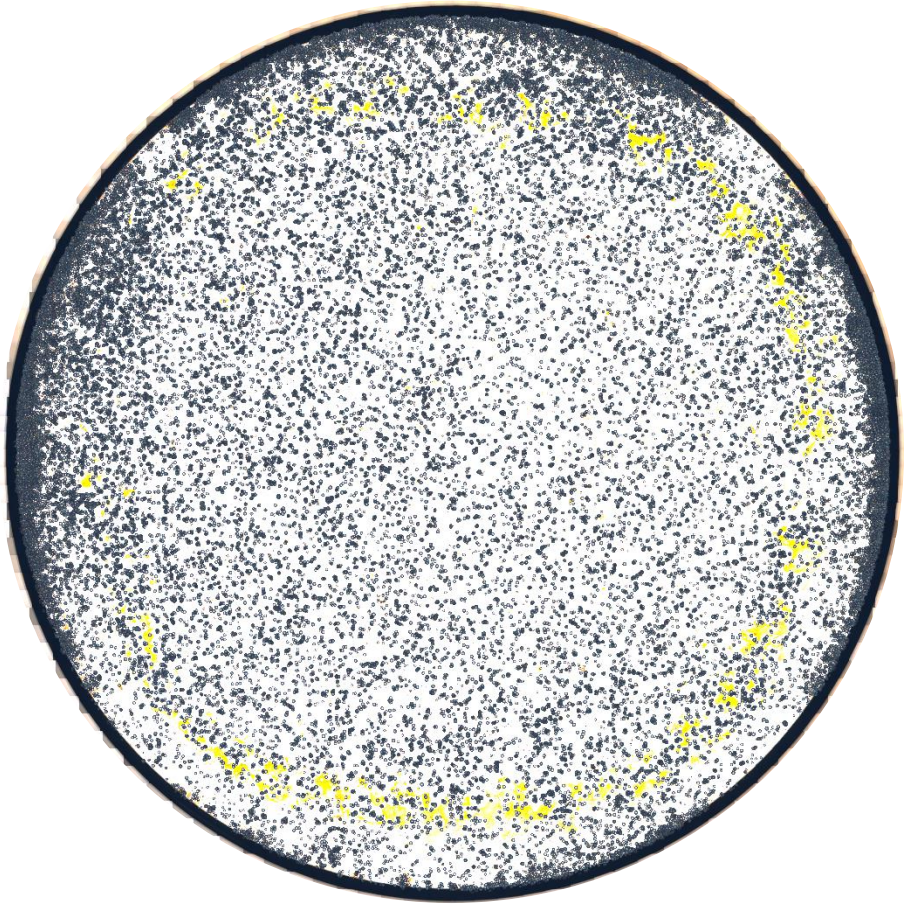
SEM
(Post-mortem)

LES Soft Cu

Anode n16

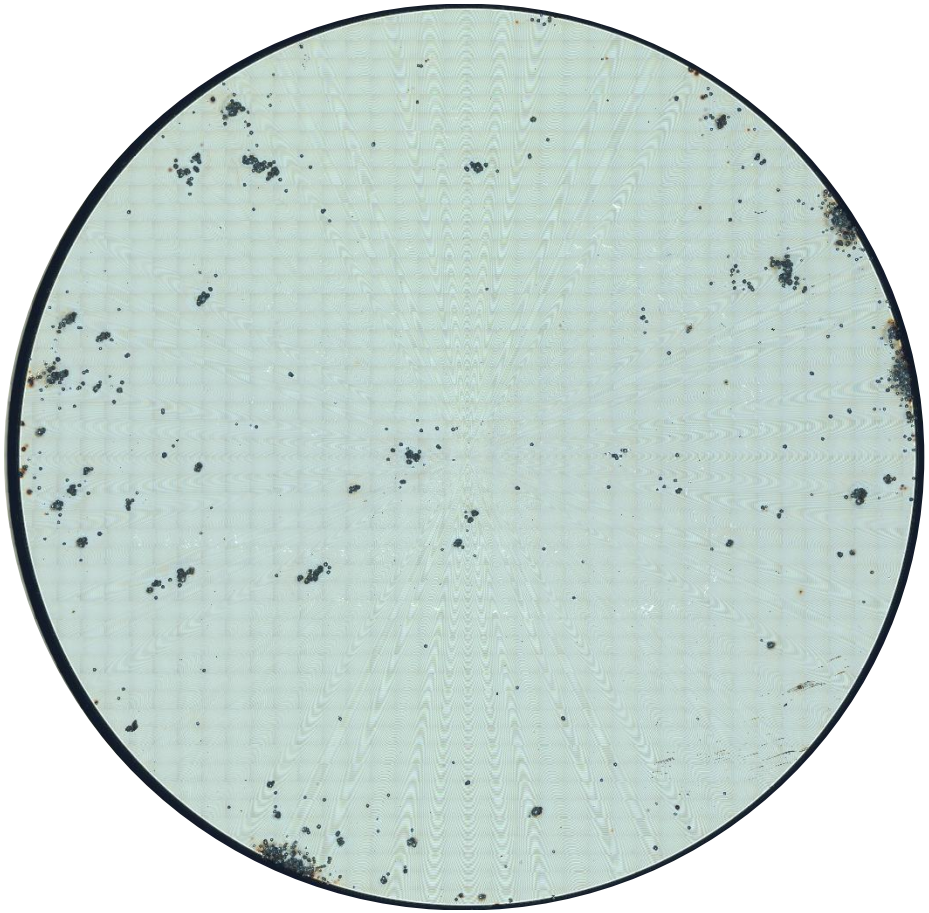


Cathode n14

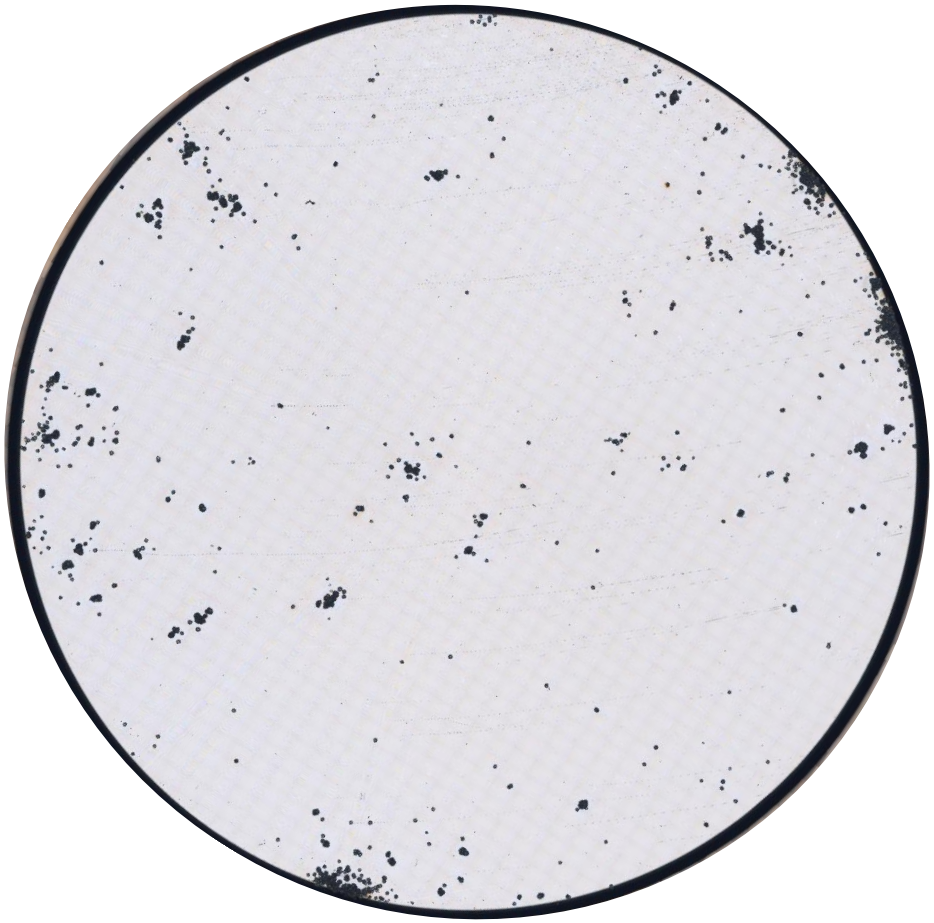


LES Hard Cu

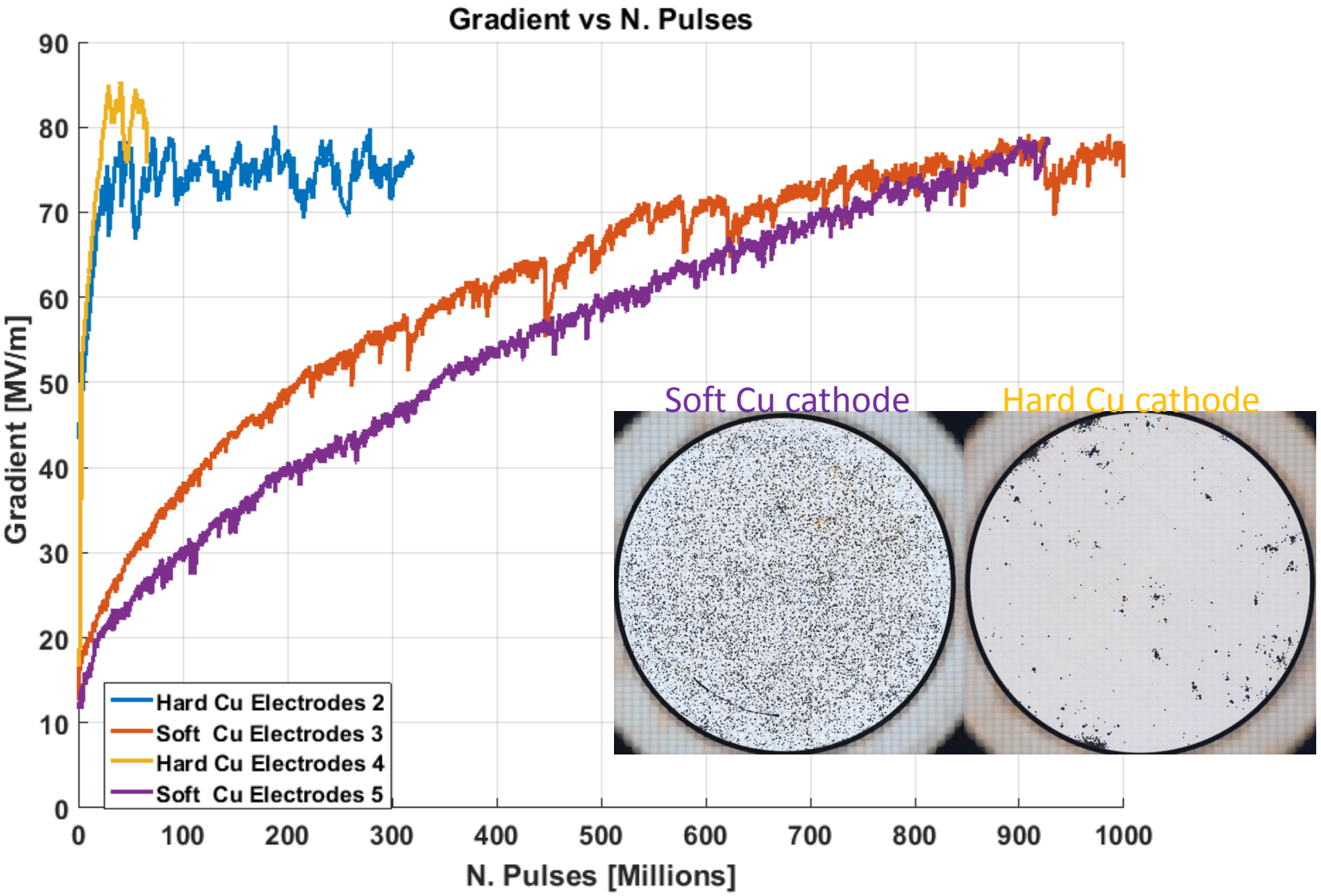
Anode hard Cu



Cathode hard Cu



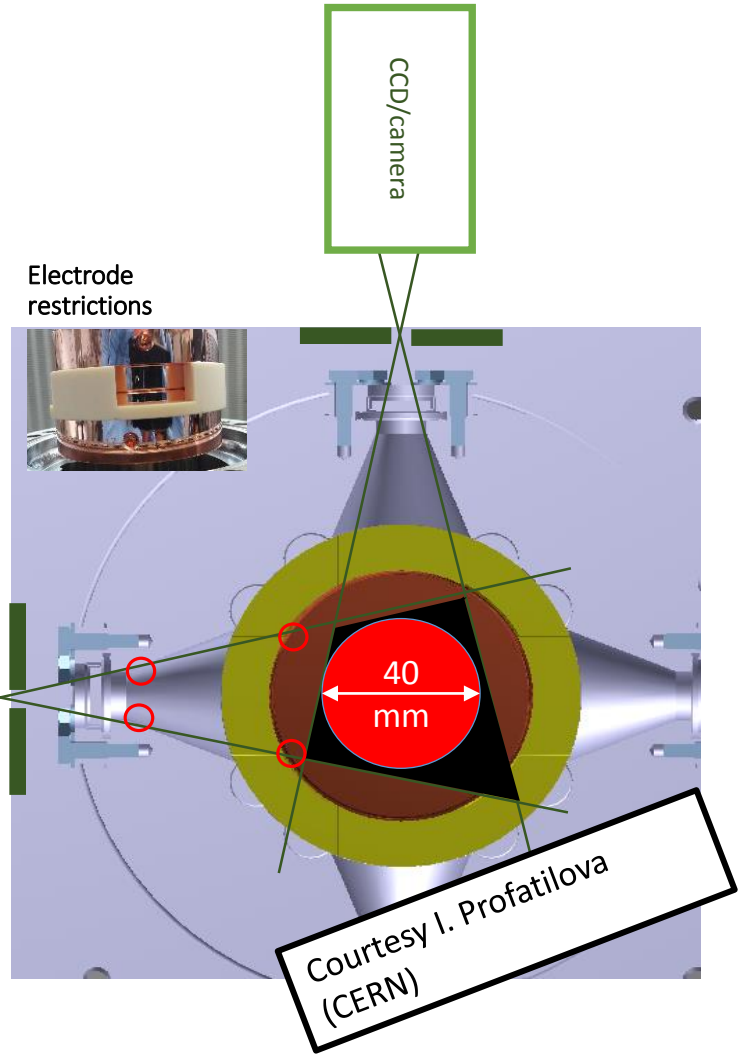
LES Hard Cu vs LES Soft Cu



Breakdown localization technique

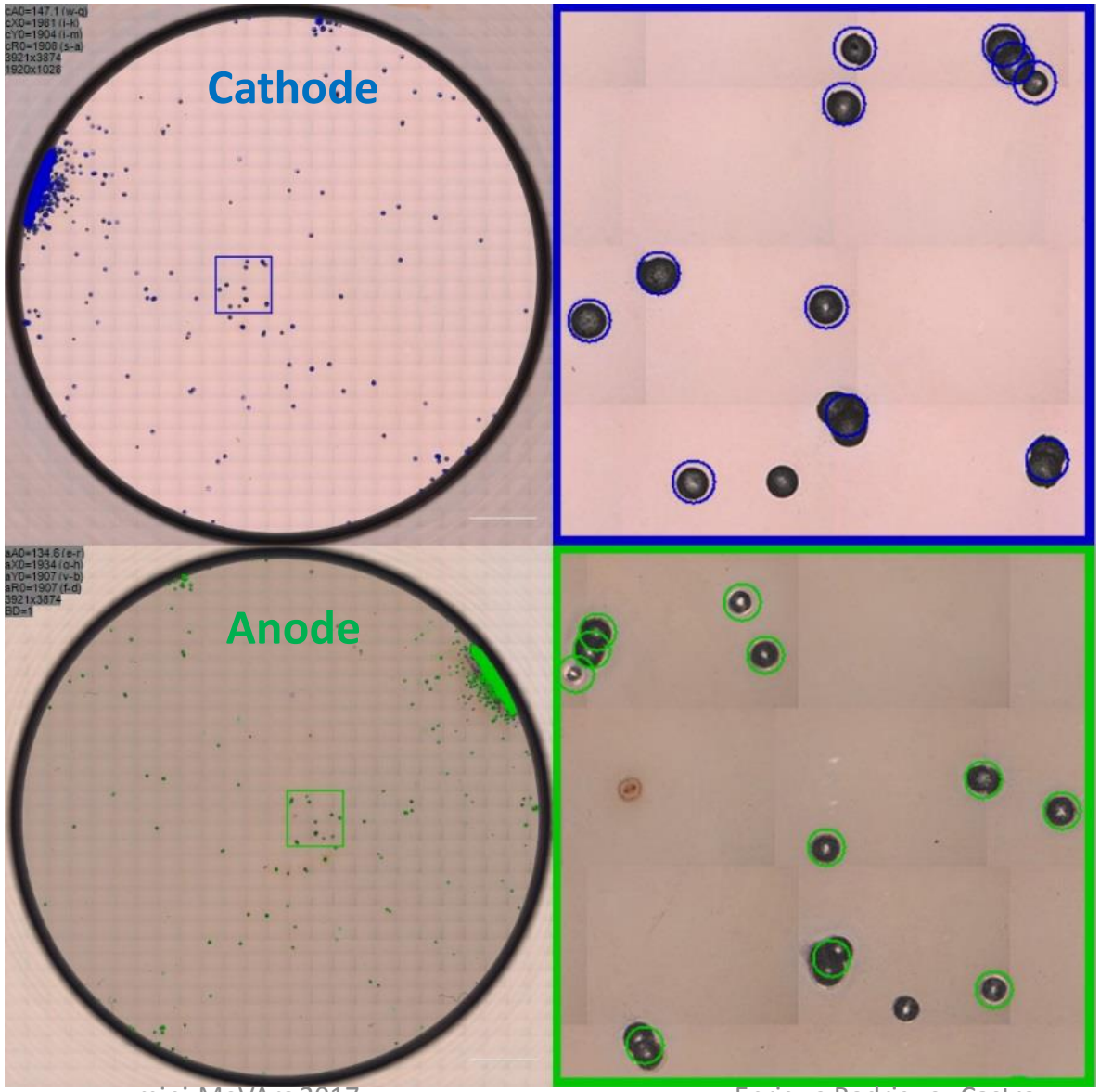
Motivation:

- ❑ find the position of BDs
- ❑ find how crater after BD relates to the next BD location.



More information about technique could be found from Xavier's Stragier slides here: http://indico.cern.ch/event/527301/

BD localization



The main data from test:
Number of breakdowns detected by Marx generator: 5690.
Number of breakdowns detected by cameras: 5665.
Difference in data: ~0.5%.

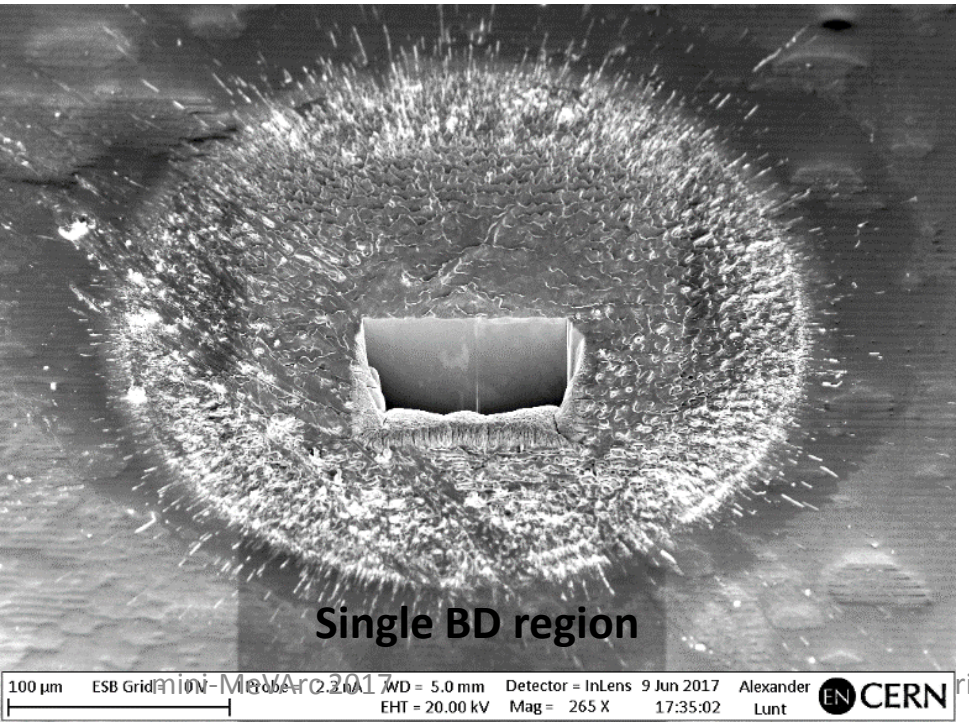
Conclusion
It works!

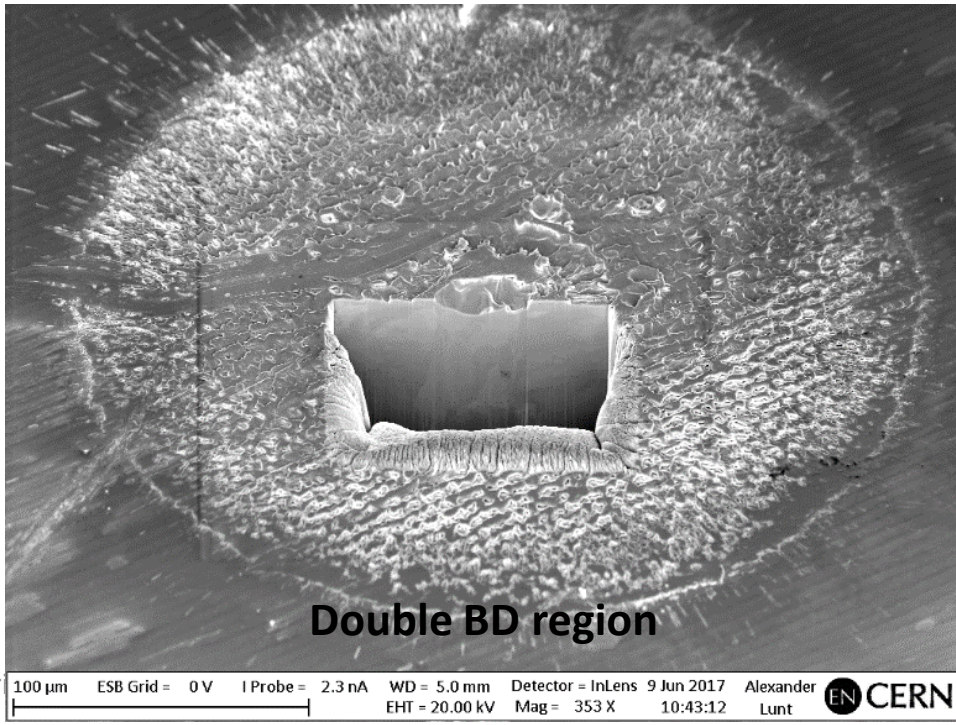
The images of hard Cu (as-machined) electrodes from optical microscope together with data from cameras.

Courtesy I. Profatilova (CERN)

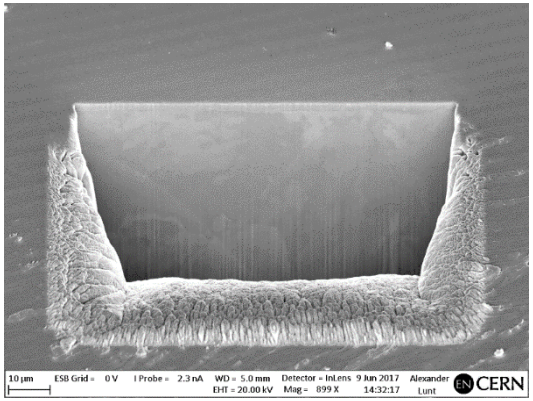
LES Post-Mortem

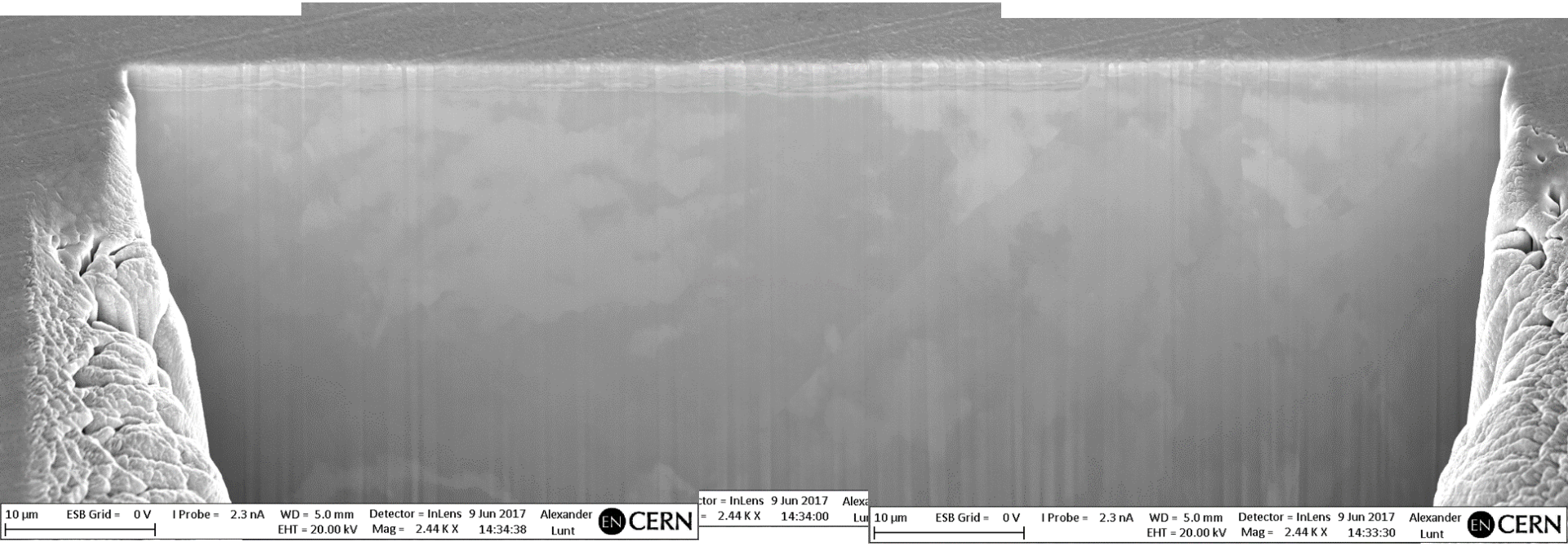
- This system allow us to FIB in well know regions
  - Reference surface → surface that has not suffer a BD
  - BD site → surface that has suffer a BD
  - Follow up site → surface that has suffer multiple BD





Cross-section: Hard Cu Reference





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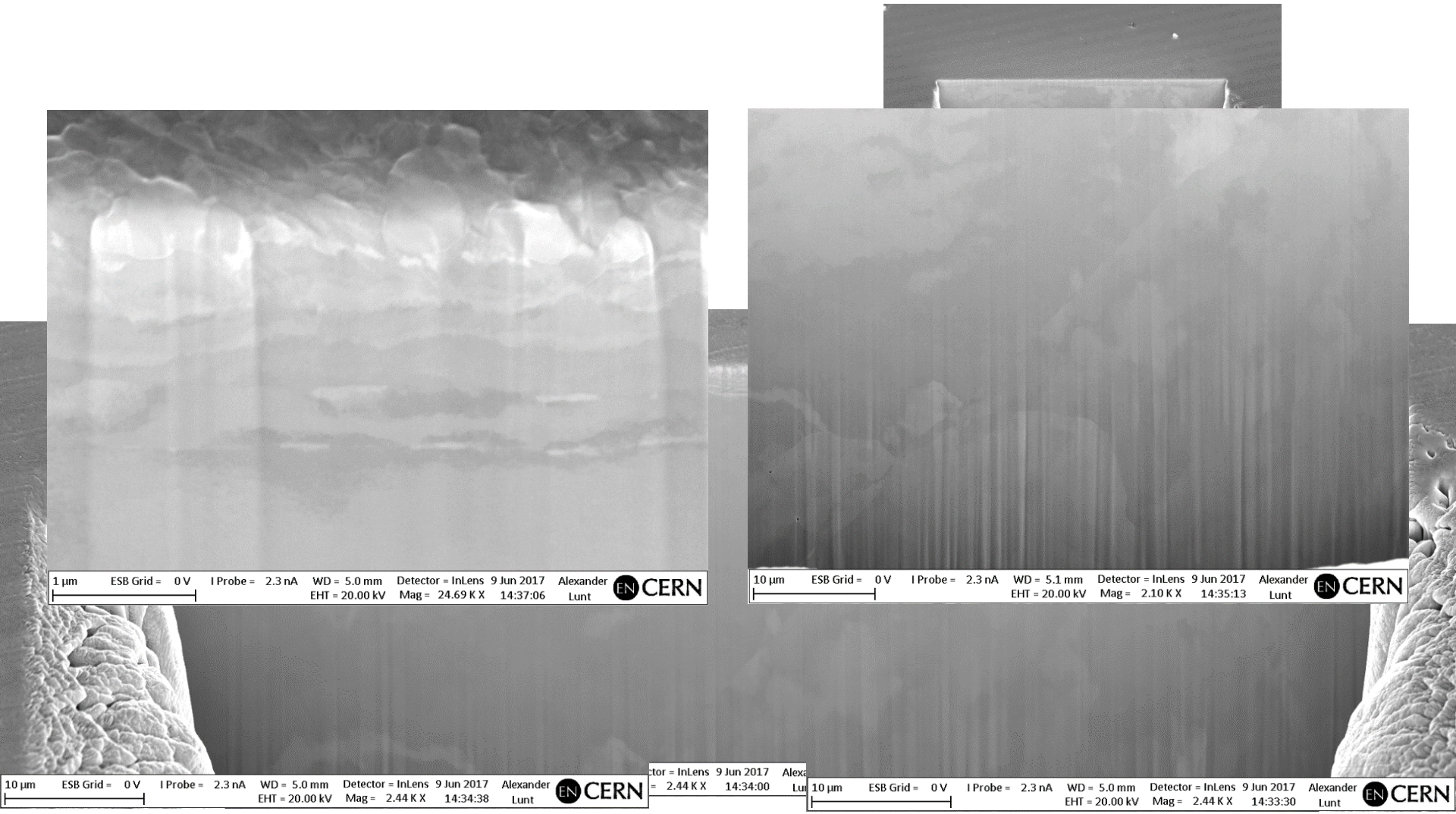

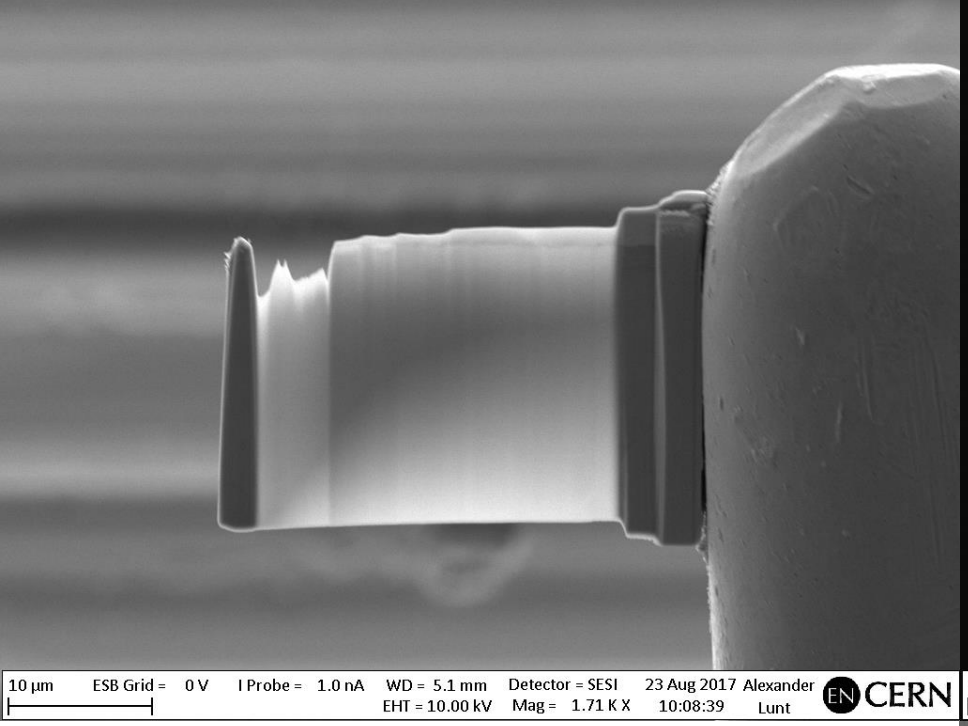

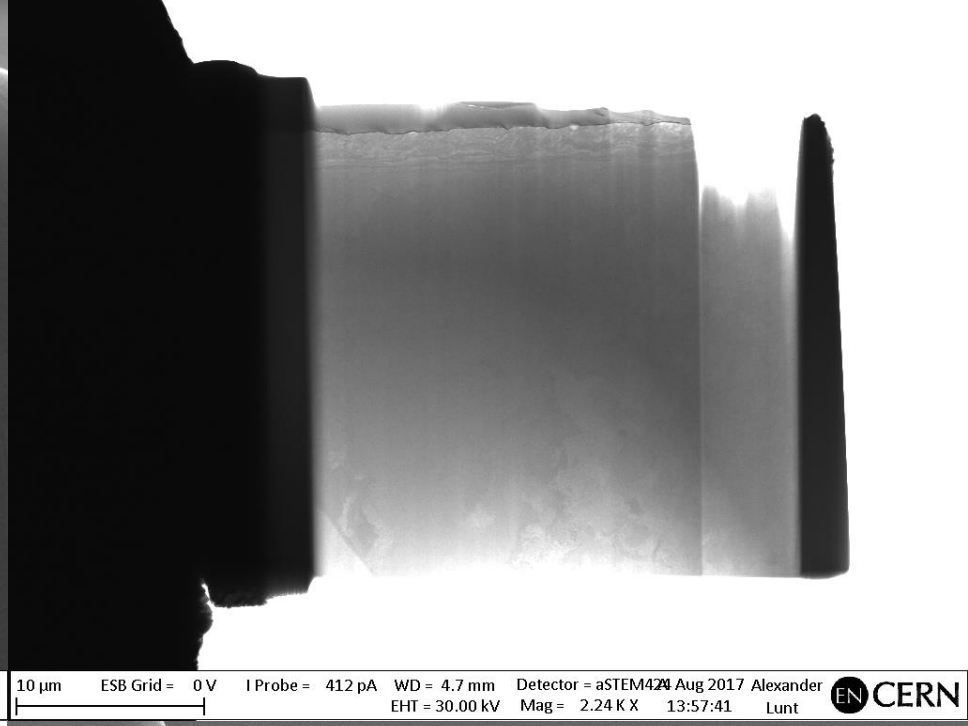

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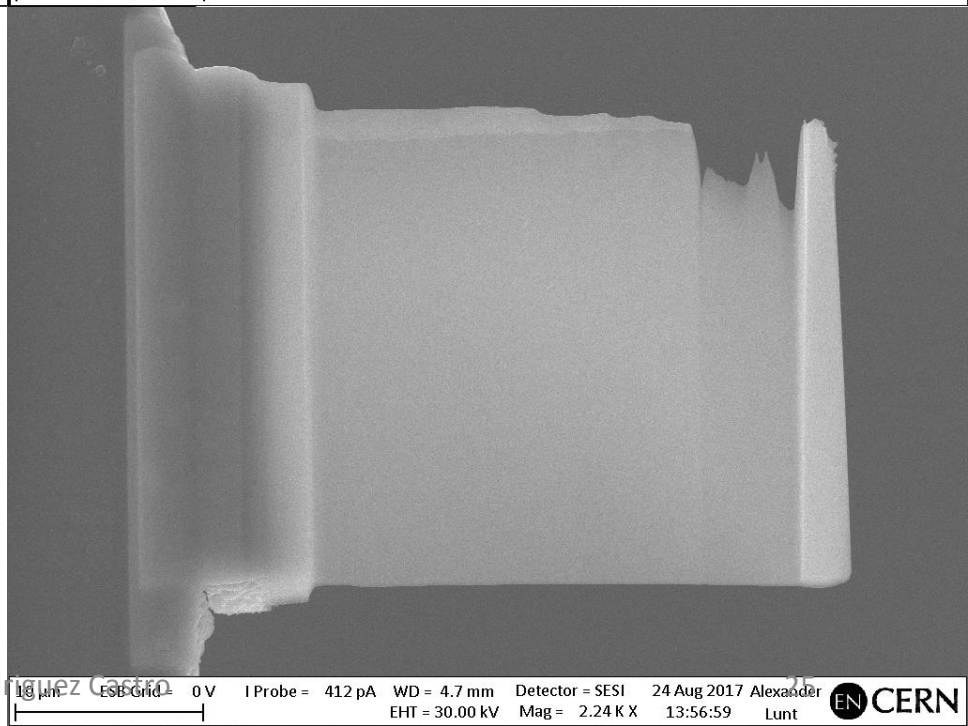
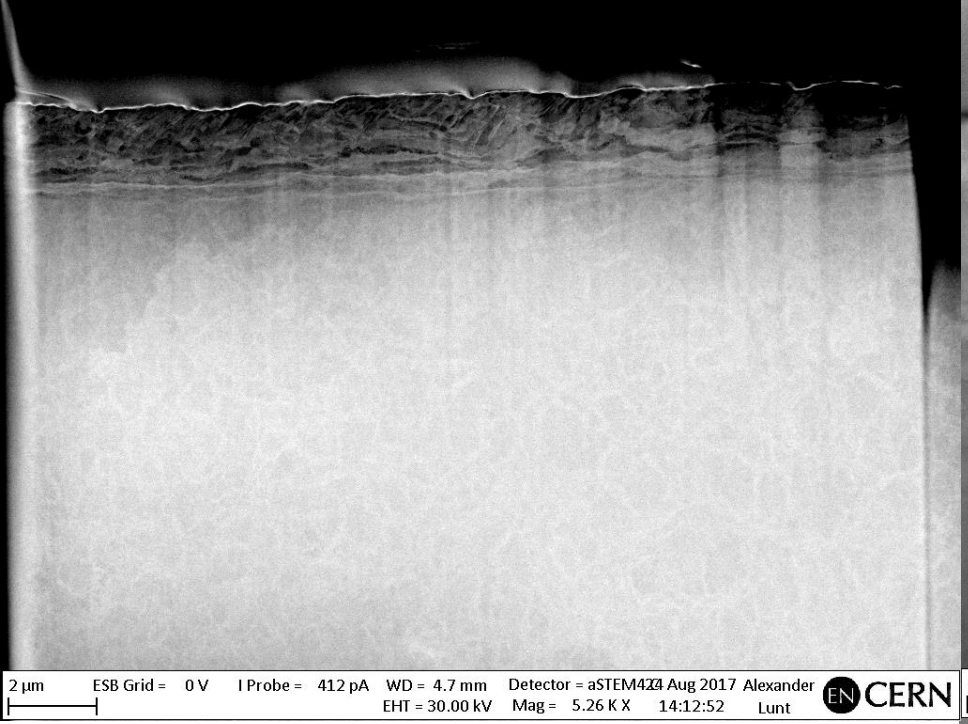
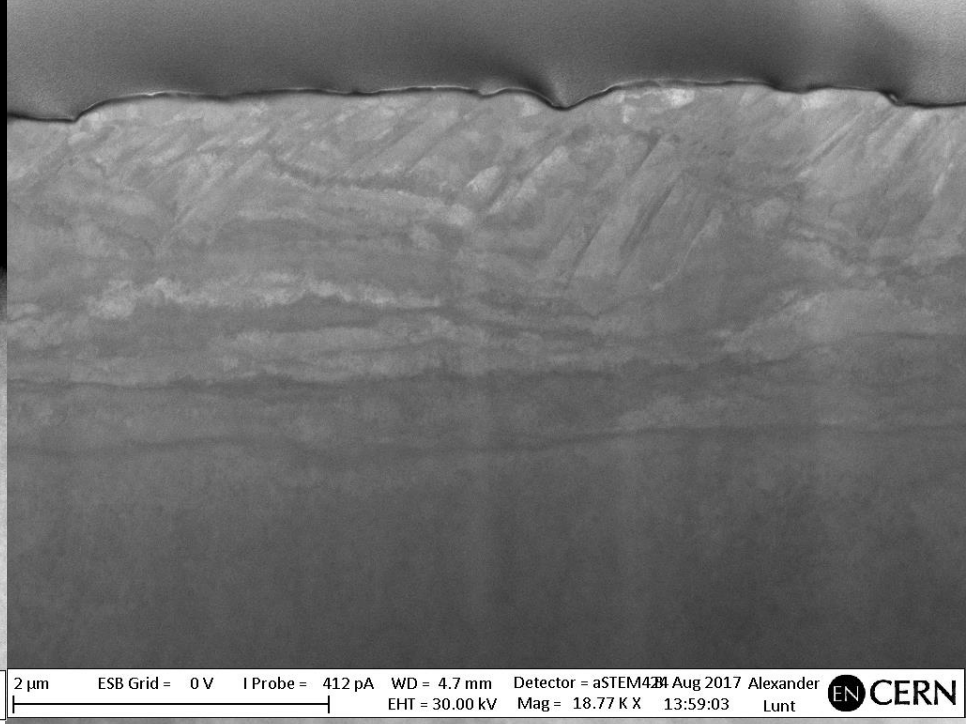
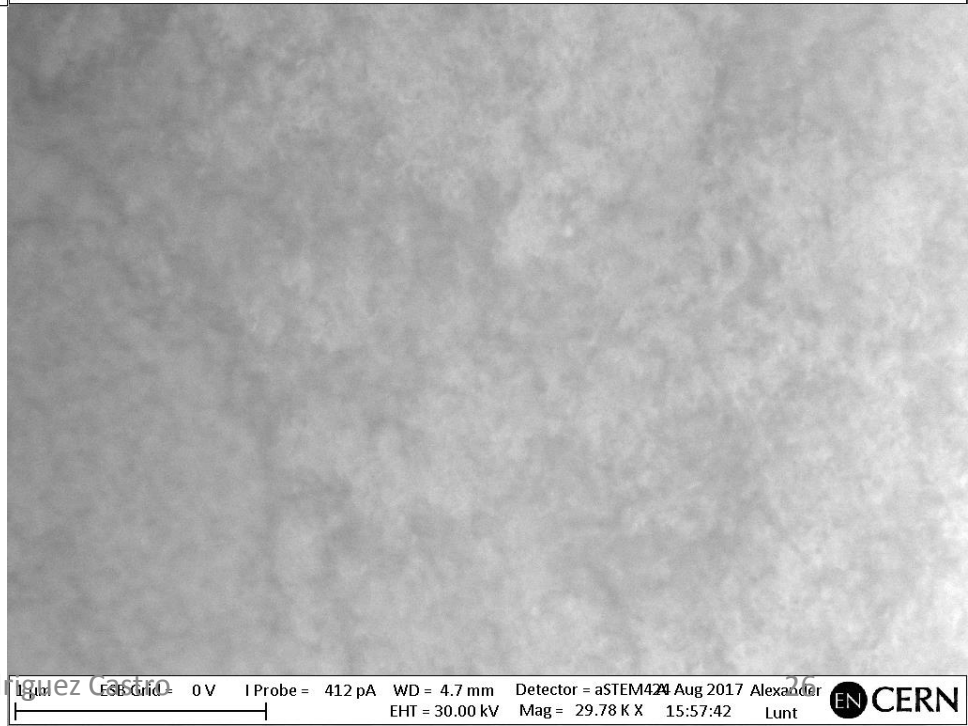


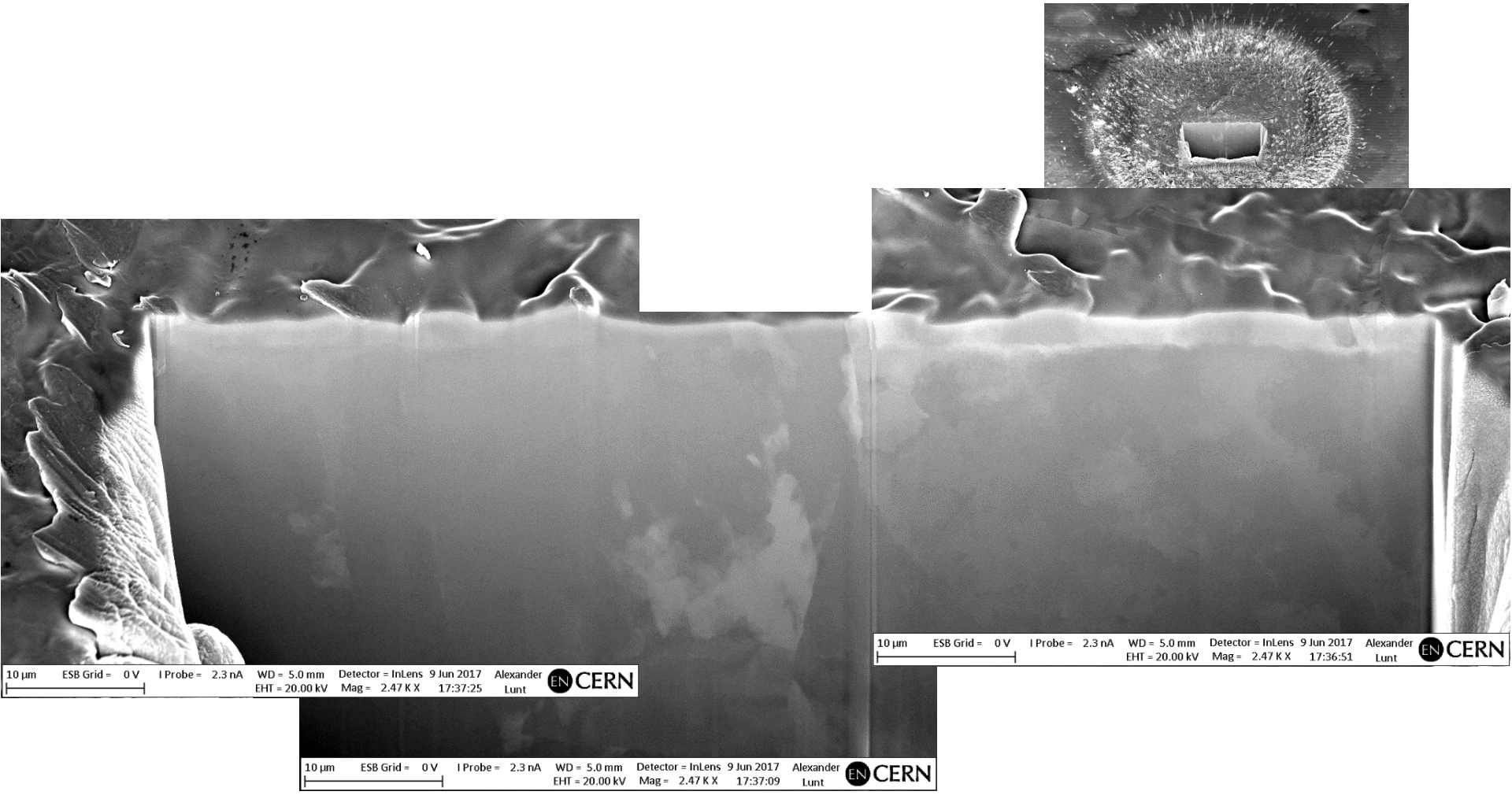




Hard Cu Reference



Cross-section: Hard Cu 1 BD



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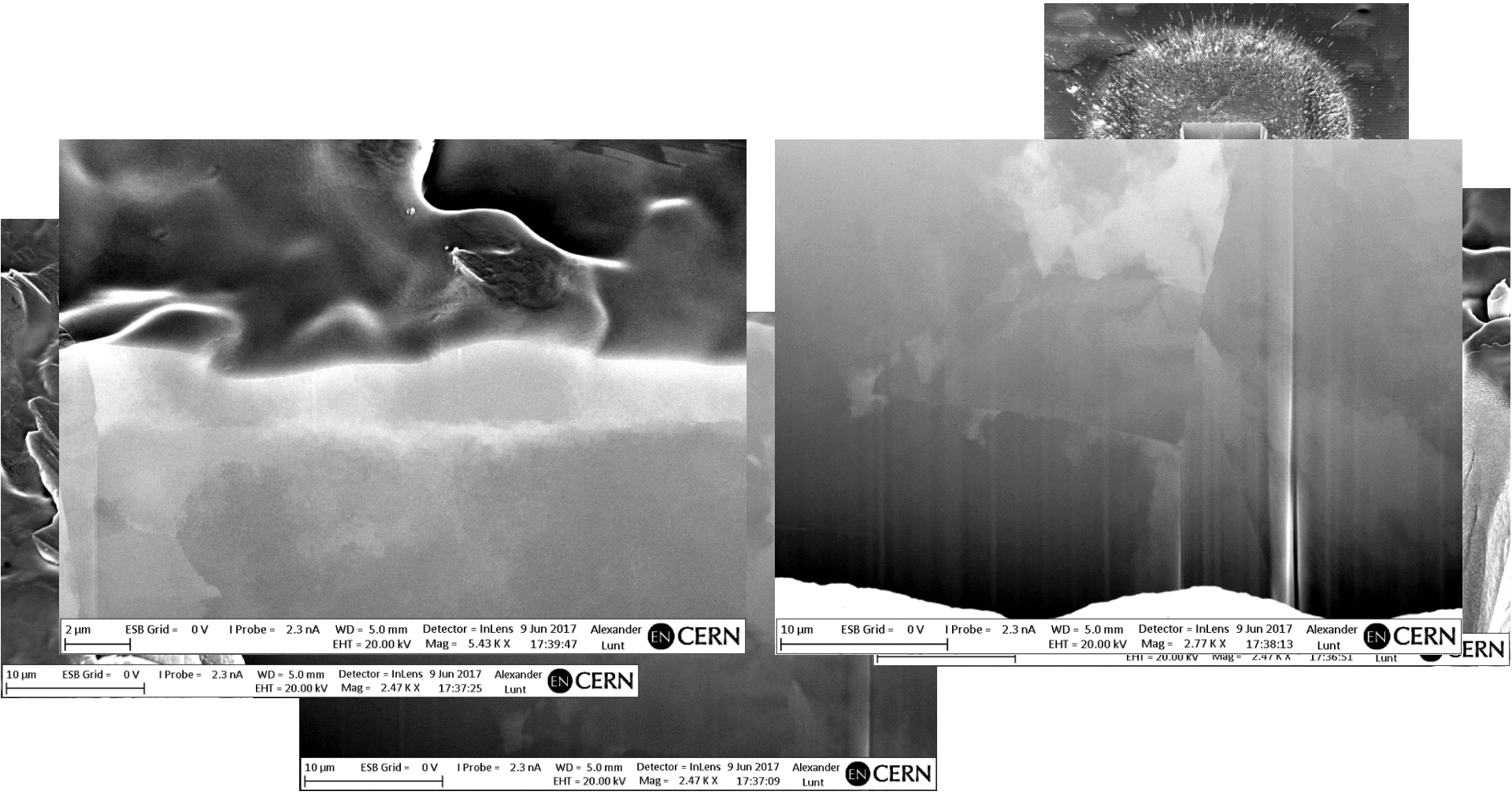
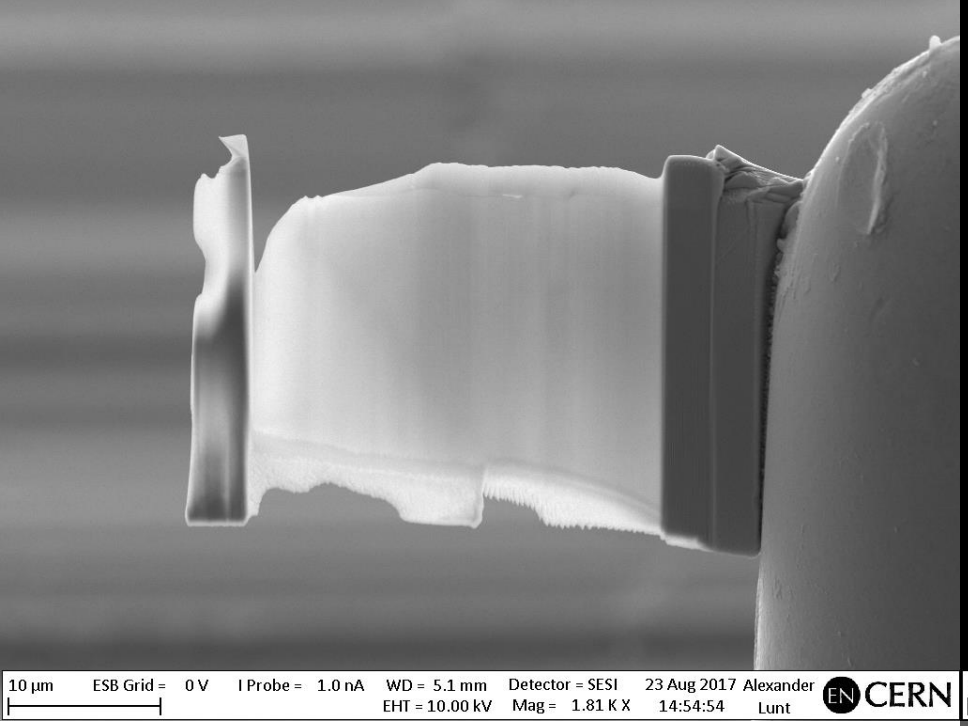
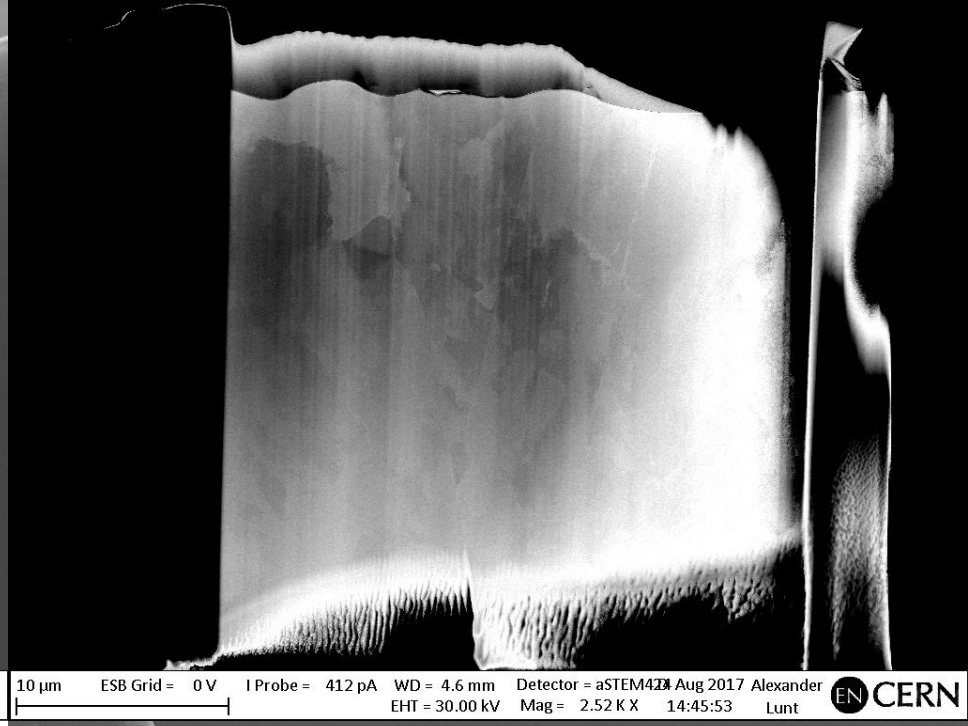
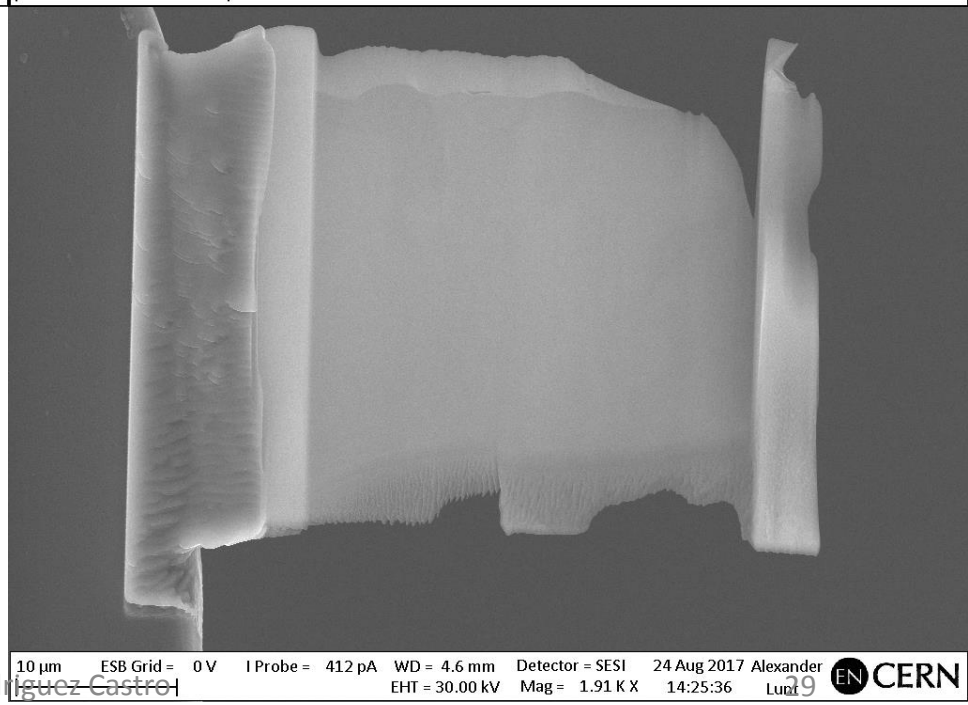
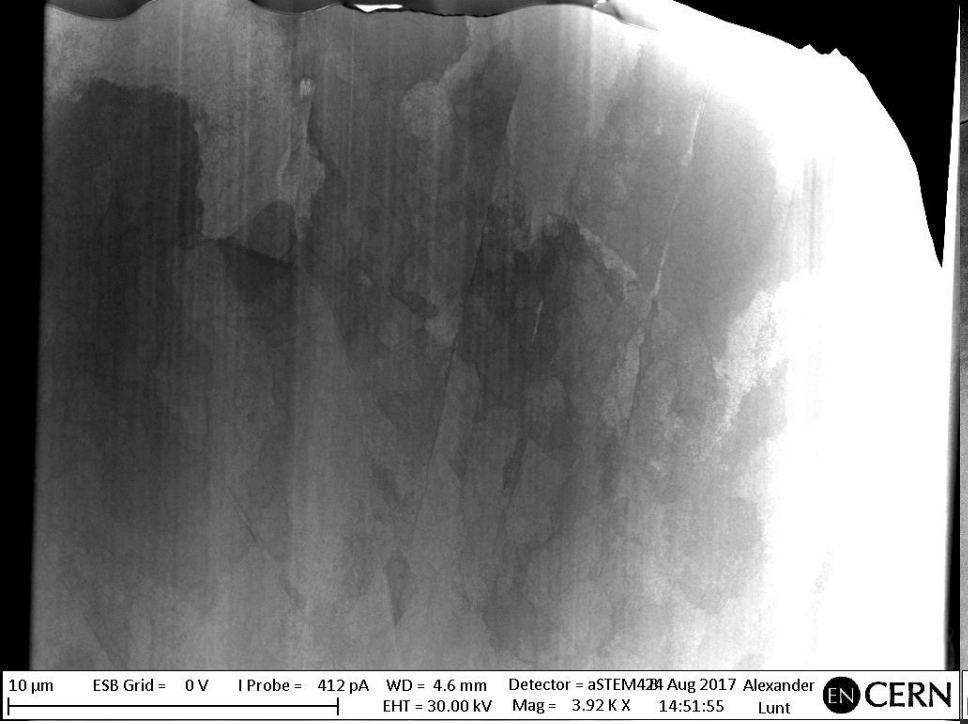


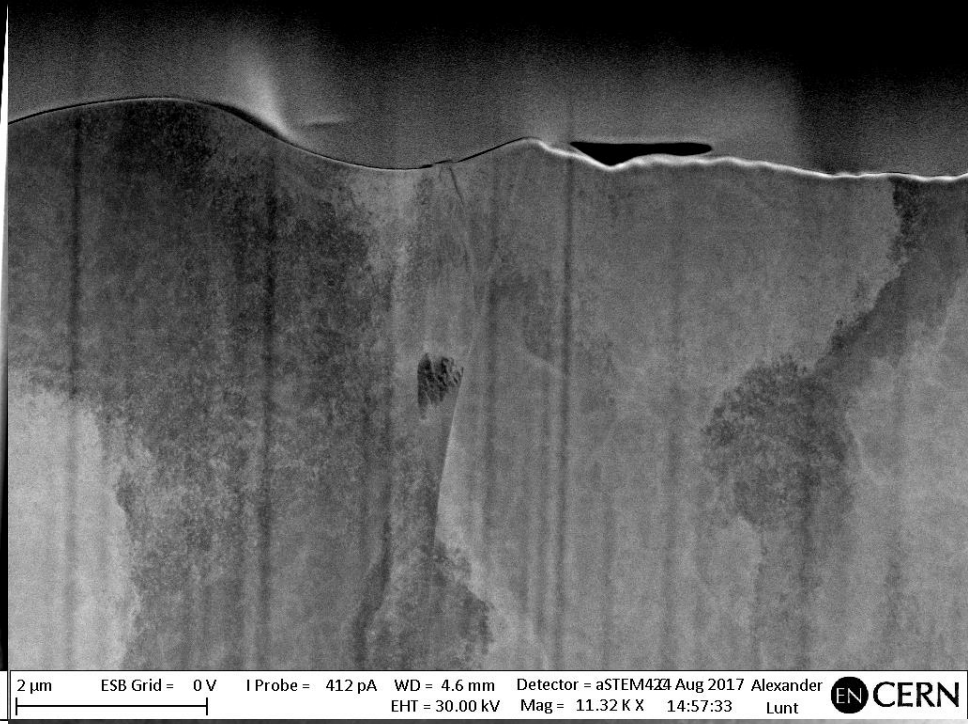

10 µm ESB Grid = 0 V I Probe = 1.0 nA WD = 5.1 mm Detector = SESI 23 Aug 2017 Alexander
EHT = 10.00 kV Mag = 1.81 K X 14:54:54 Lunt
CERN


10 µm ESB Grid = 0 V I Probe = 412 pA WD = 4.6 mm Detector = aSTEM420 24 Aug 2017 Alexander
EHT = 30.00 kV Mag = 2.52 K X 14:45:53 Lunt
CERN

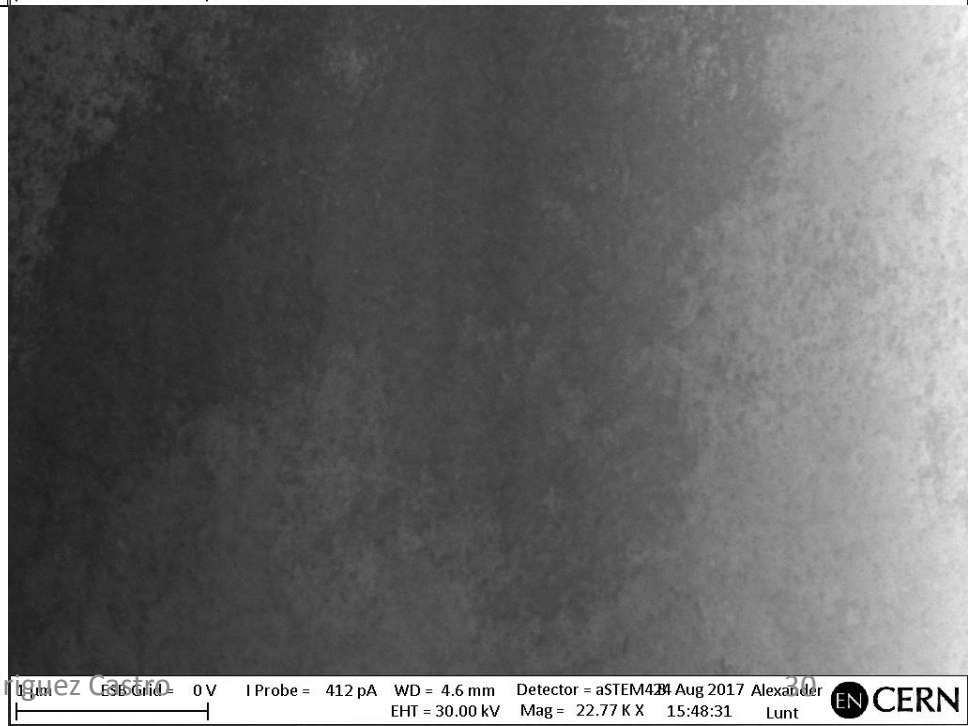
Hard Cu 1 BD


10 µm ESB Grid = 0 V I Probe = 412 pA WD = 4.6 mm Detector = SESI 24 Aug 2017 Alexander
EHT = 30.00 kV Mag = 1.91 K X 14:25:36 Lunt
CERN

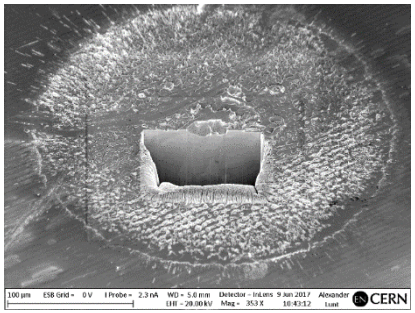

10 µm ESB Grid = 0 V I Probe = 412 pA WD = 4.6 mm Detector = aSTEM4 24 Aug 2017 Alexander
EHT = 30.00 kV Mag = 3.92 K X 14:51:55 Lunt
CERN

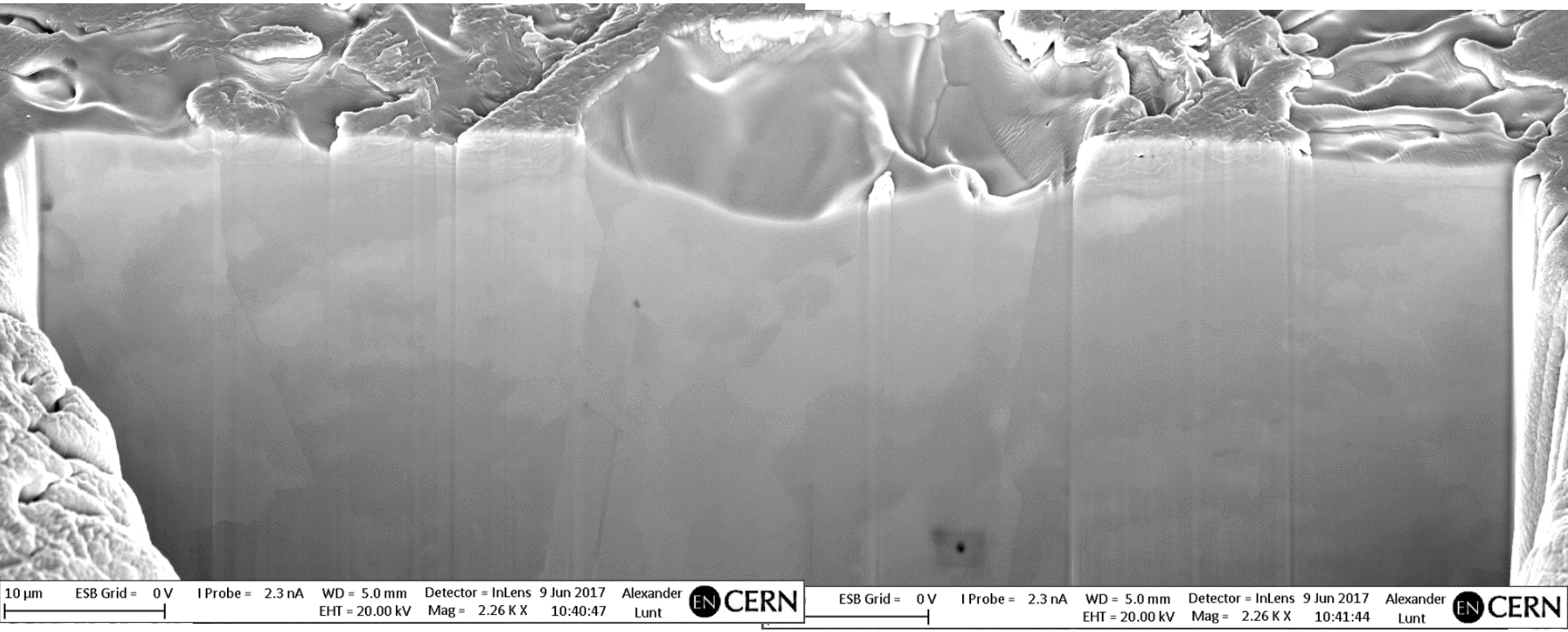

2 µm ESB Grid = 0 V I Probe = 412 pA WD = 4.6 mm Detector = aSTEM4 24 Aug 2017 Alexander
EHT = 30.00 kV Mag = 11.32 K X 14:57:33 Lunt
CERN

Hard Cu 1 BD

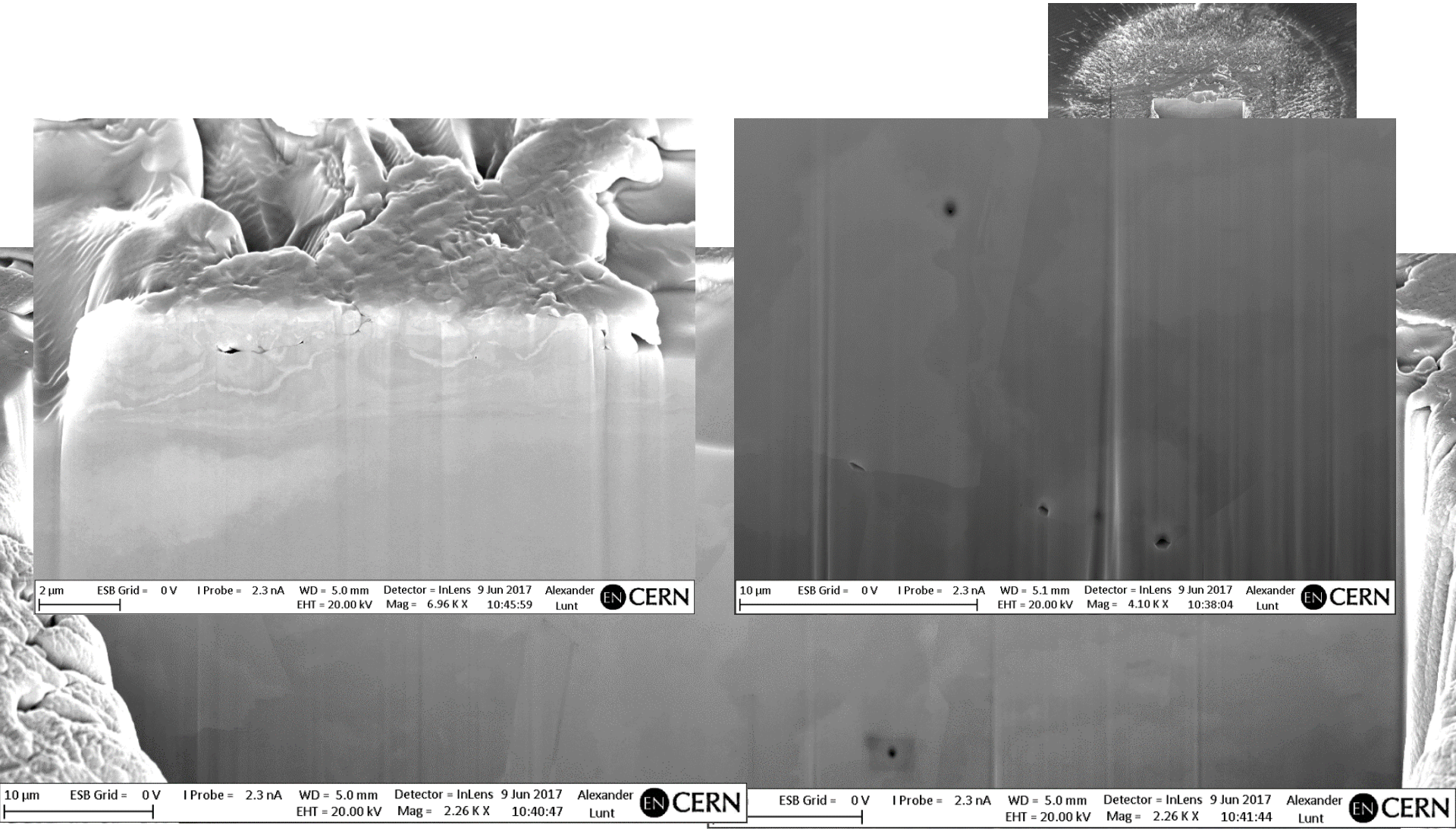

1 µm ESB Grid = 0 V I Probe = 412 pA WD = 4.6 mm Detector = aSTEM4 24 Aug 2017 Alexander
EHT = 30.00 kV Mag = 22.77 K X 15:48:31 Lunt
CERN

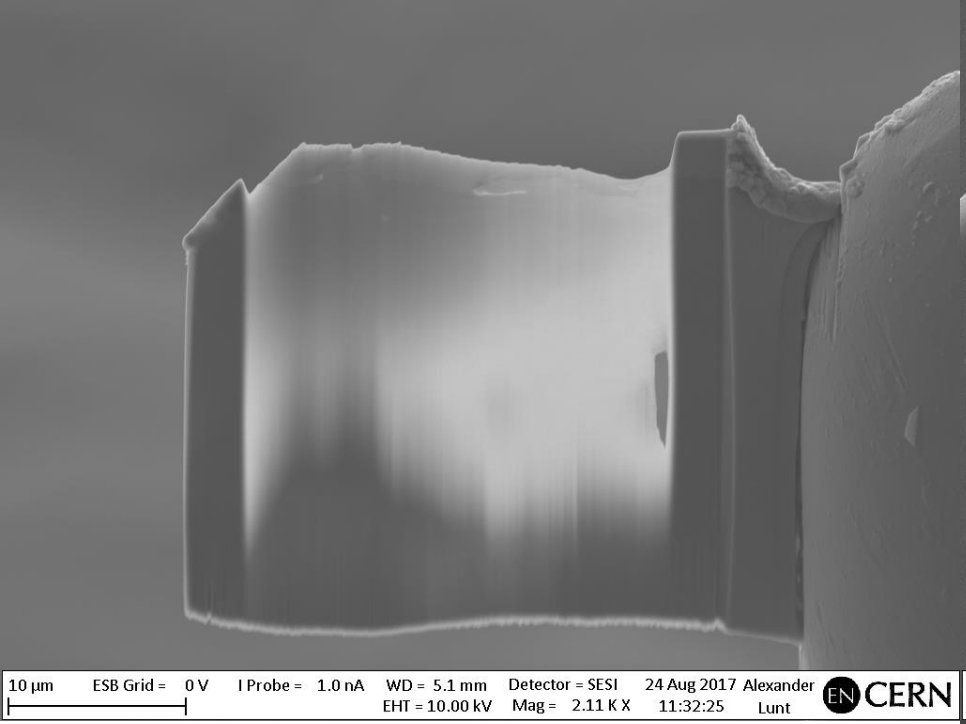
Cross-section: Hard Cu 2 BD

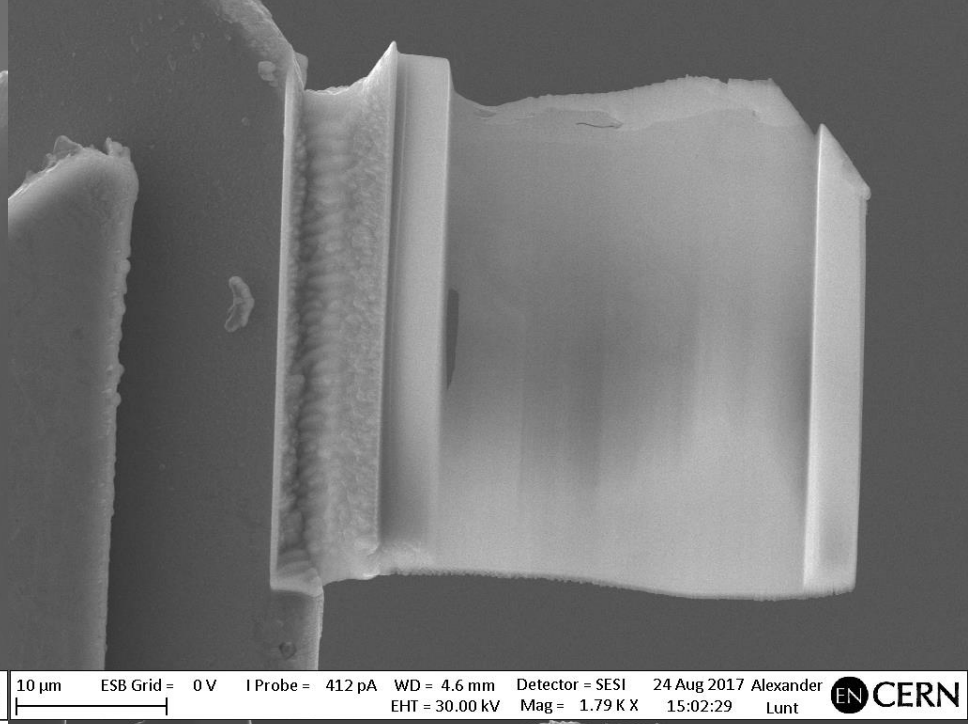




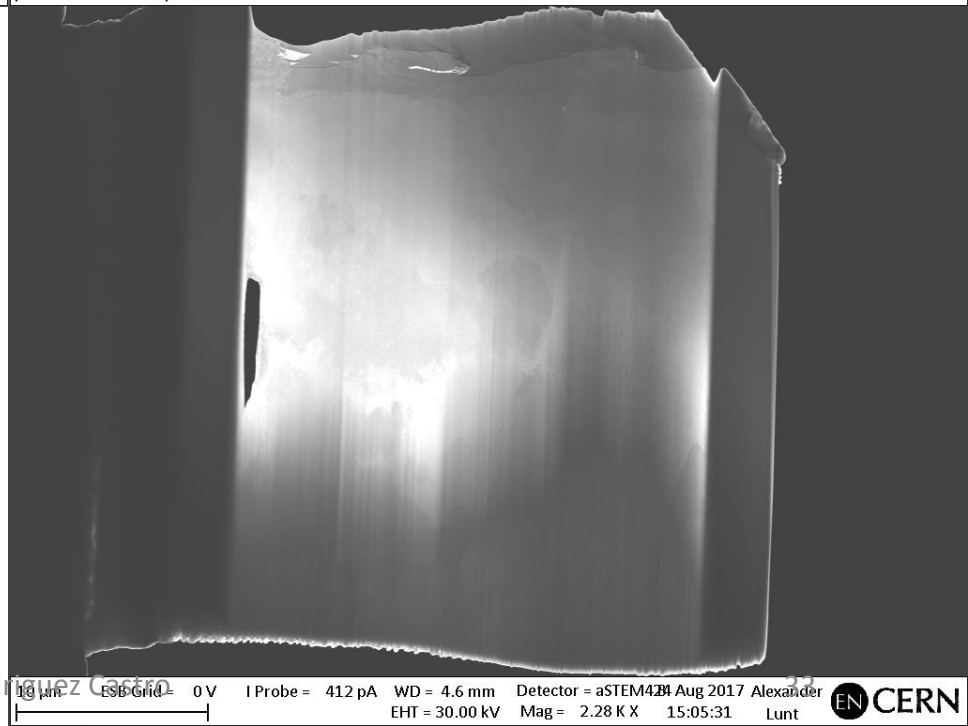
Cross-section: Hard Cu 2 BD




10 µm ESB Grid = 0 V I Probe = 1.0 nA WD = 5.1 mm Detector = SESI 24 Aug 2017 Alexander
EHT = 10.00 kV Mag = 2.11 K X 11:32:25 Lunt
CERN

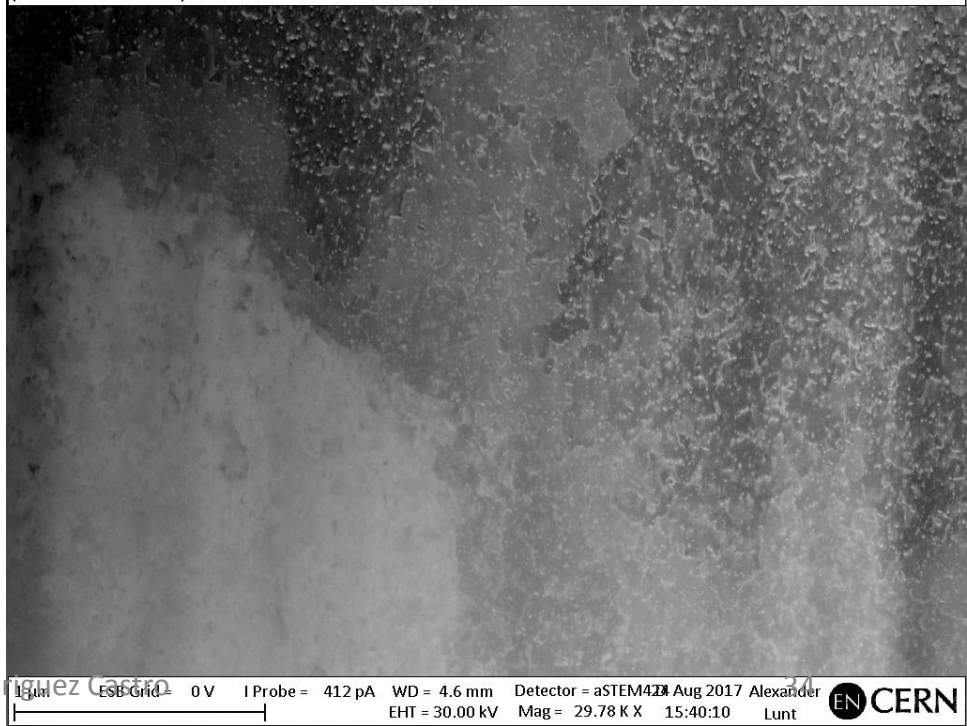

10 µm ESB Grid = 0 V I Probe = 412 pA WD = 4.6 mm Detector = SESI 24 Aug 2017 Alexander
EHT = 30.00 kV Mag = 1.79 K X 15:02:29 Lunt
CERN

Hard Cu 2 BD

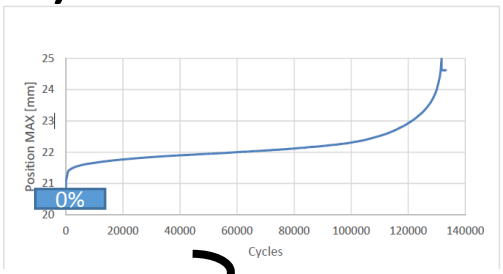

10 µm ESB Grid = 0 V I Probe = 412 pA WD = 4.6 mm Detector = aSTEM4 24 Aug 2017 Alexander
EHT = 30.00 kV Mag = 2.28 K X 15:05:31 Lunt
CERN

Hard Cu 2 BD


2 µm ESB Grid = 0 V I Probe = 412 pA WD = 4.6 mm EHT = 30.00 kV
Detector = aSTEM4 Mag = 9.99 K X
24 Aug 2017 15:09:37
Alexander Lunt
CERN

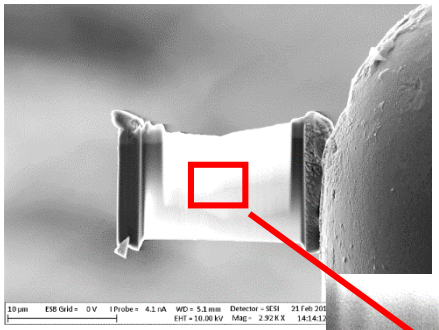

1 µm ESB Grid = 0 V I Probe = 412 pA WD = 4.6 mm EHT = 30.00 kV
Detector = aSTEM4 Mag = 29.78 K X
24 Aug 2017 15:40:10
Alexander Lunt
CERN

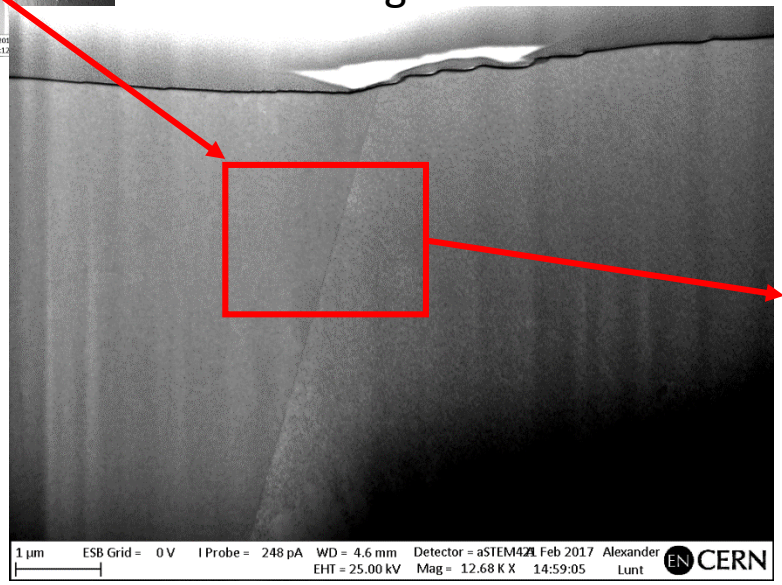
Reference (Soft Cu STEM)

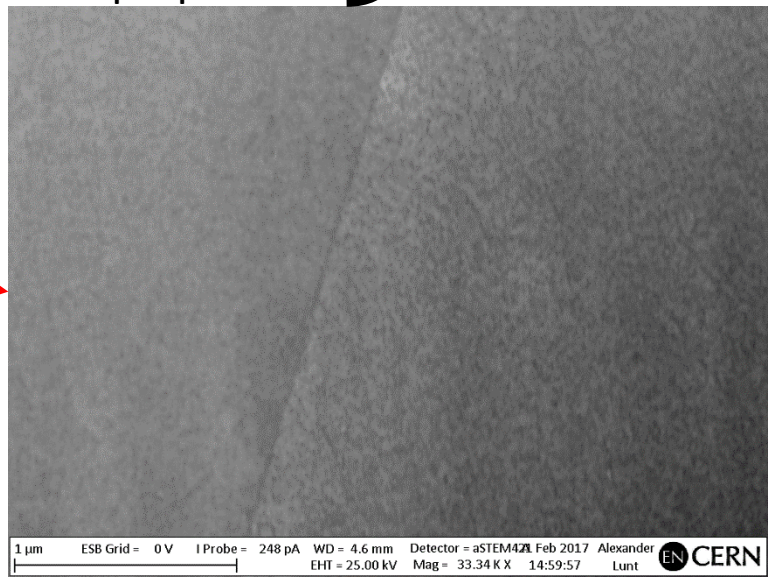


- Grain with different orientation
  - Homogeneous
  - Individual Dislocations?
  - Damage effect due to Ga beam preparation

To be confirmed by TEM

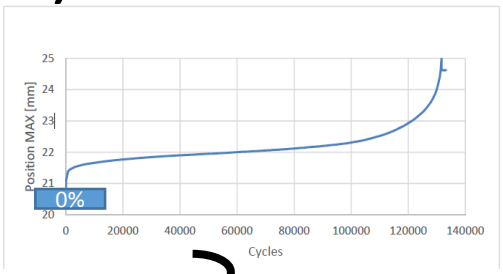






Enrique Rodriguez Castro

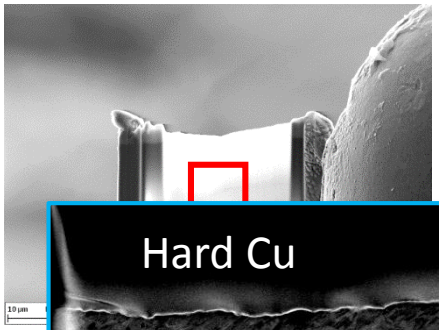
Reference (Soft Cu STEM)

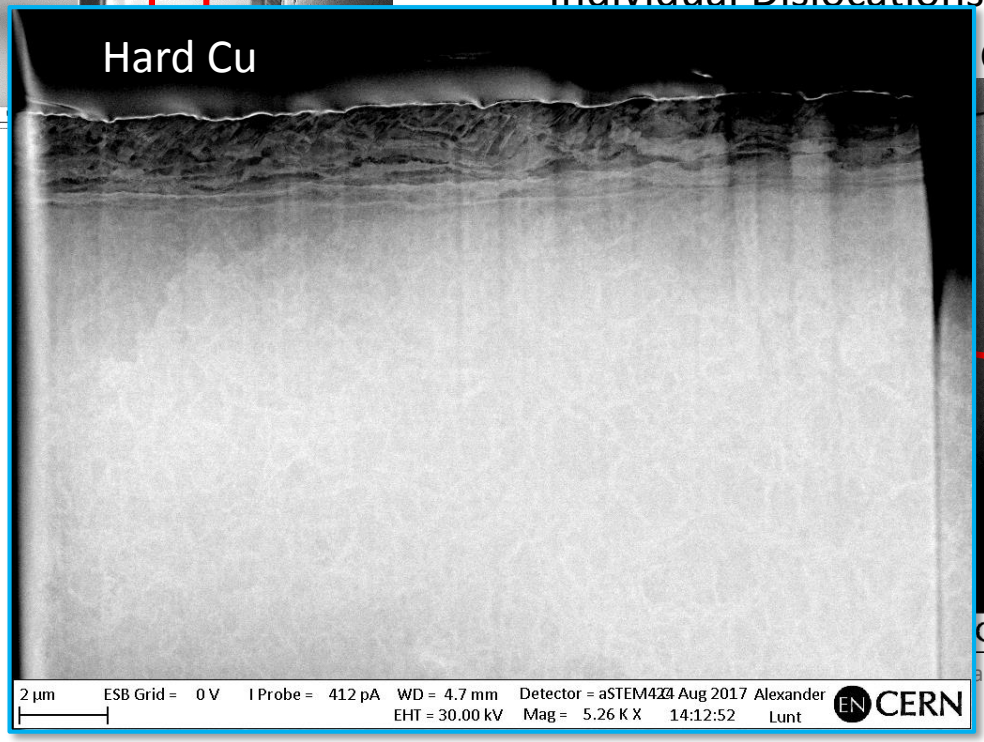


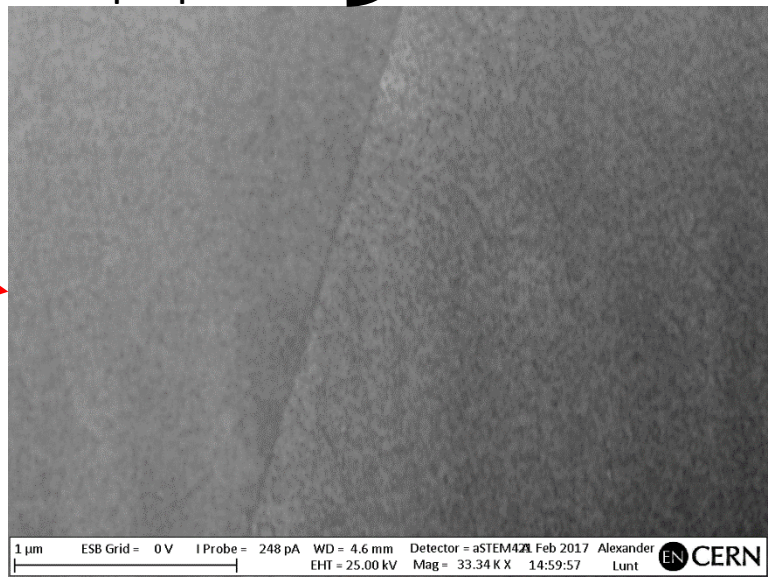
- Grain with different orientation
  - Homogeneous
  - Individual Dislocations?

To be confirmed by TEM

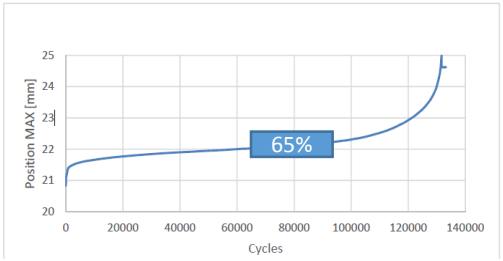
Ga beam preparation

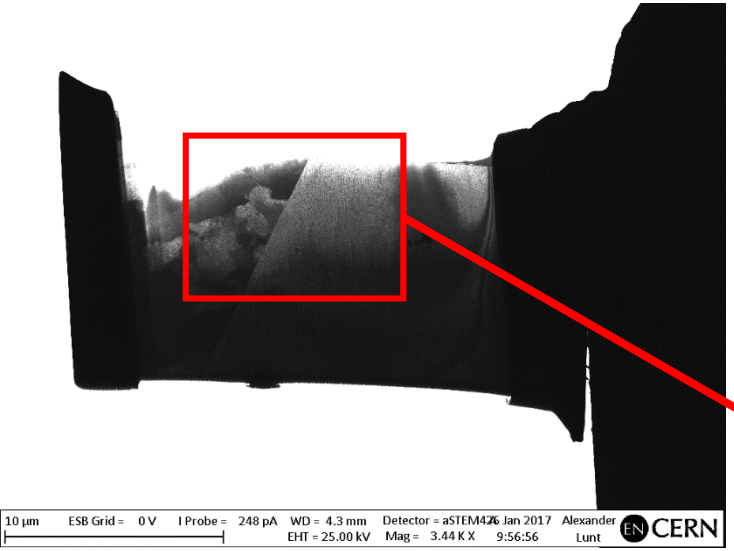


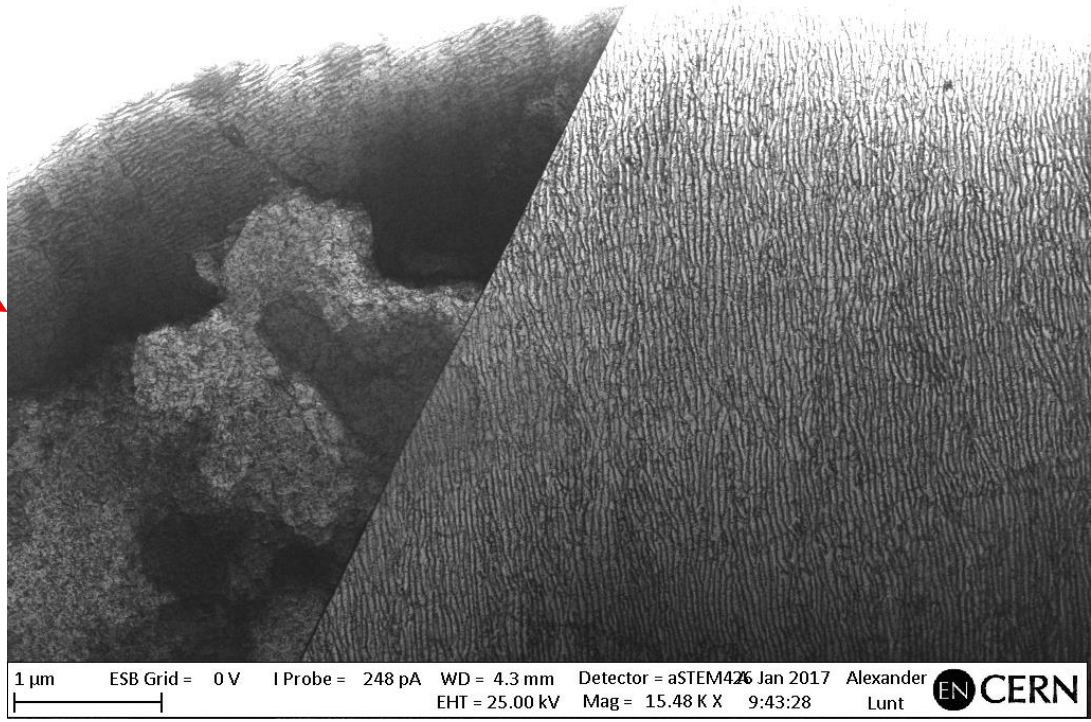




Fatigue (65% LT)

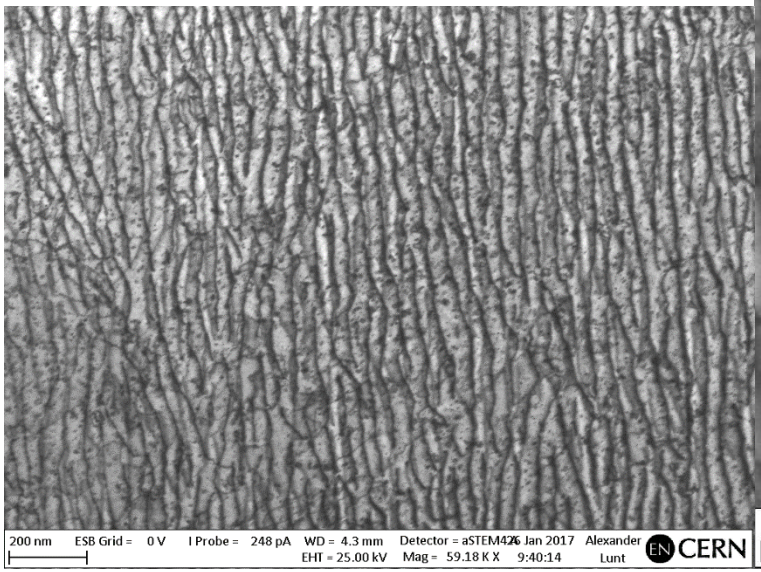


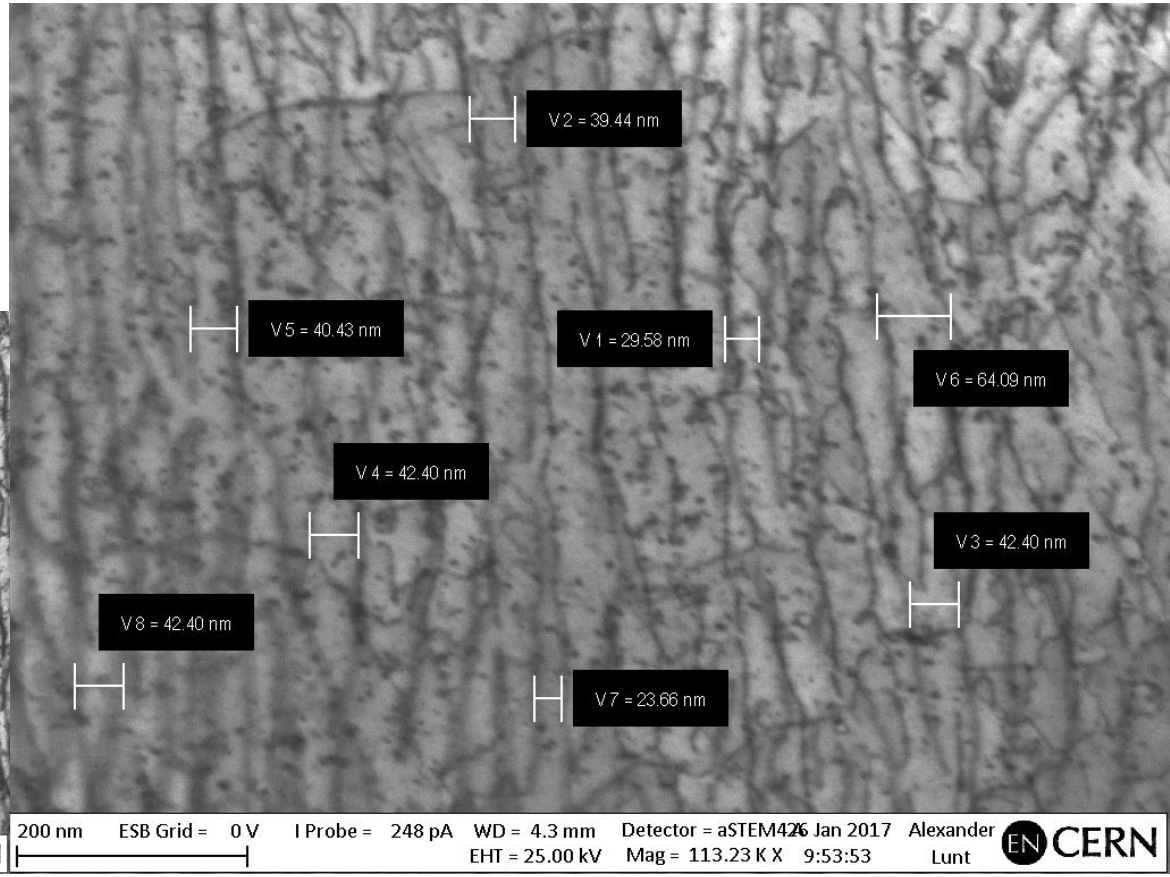




Fatigue (65% LT)

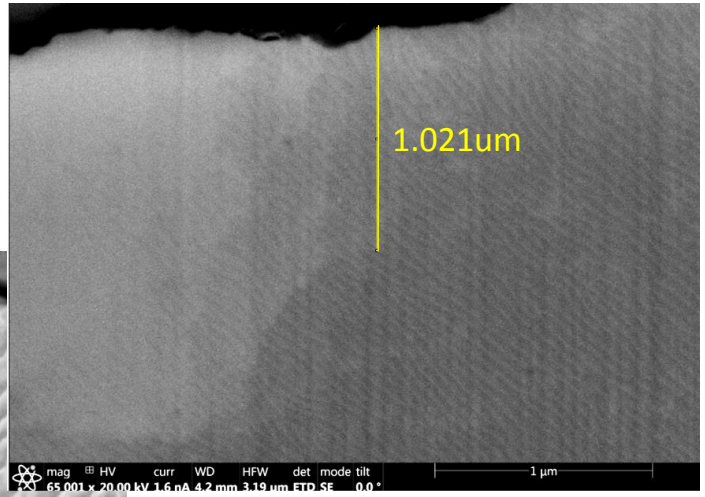
- Well structured in ladder type
- 20 – 60 nm



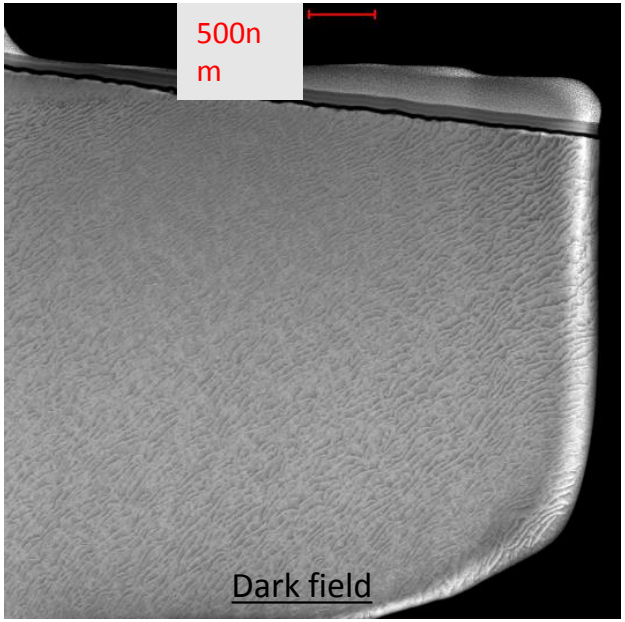


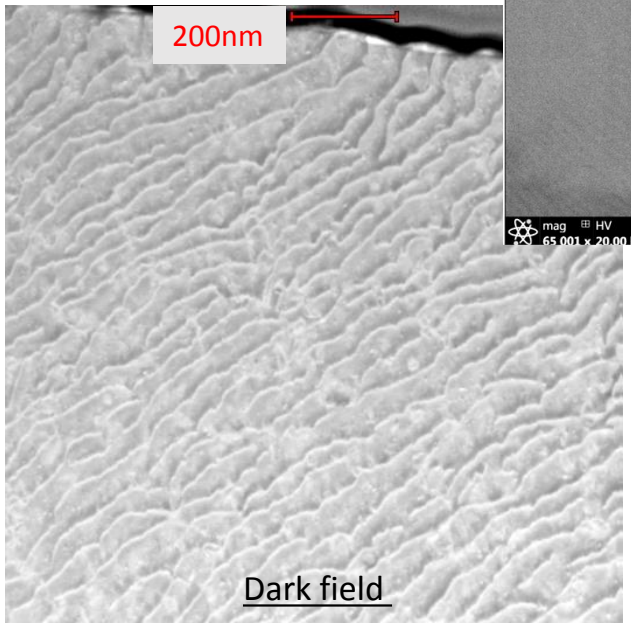
Observation on real structures

ECC image at a crater site, RF (crab-cavity)



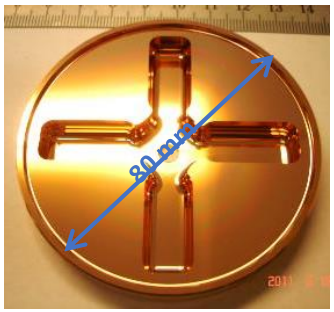
STEM image at a reference site, RF (TD24-S23)





Courtesy A. Yashar, I. Popov, Y. Ashkenazy (Hebrew University)

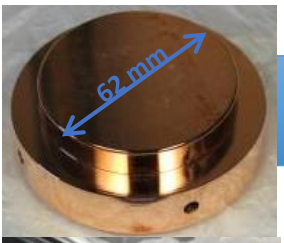
Microscopy for CLIC



UP-Diamond machining

Cleaning
Pollution analysis

SEM
(QC of the surface)

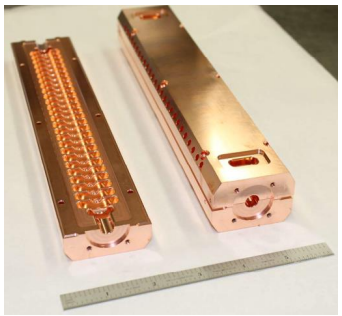


LES
HARD-Cu

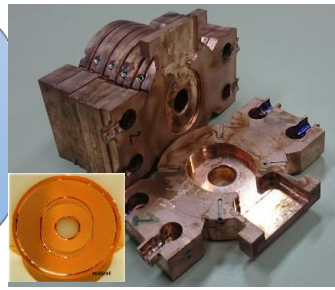
Assembly:
H_2 diffusion bonding/brazing

LES
SOFT-Cu

Vacuum baking



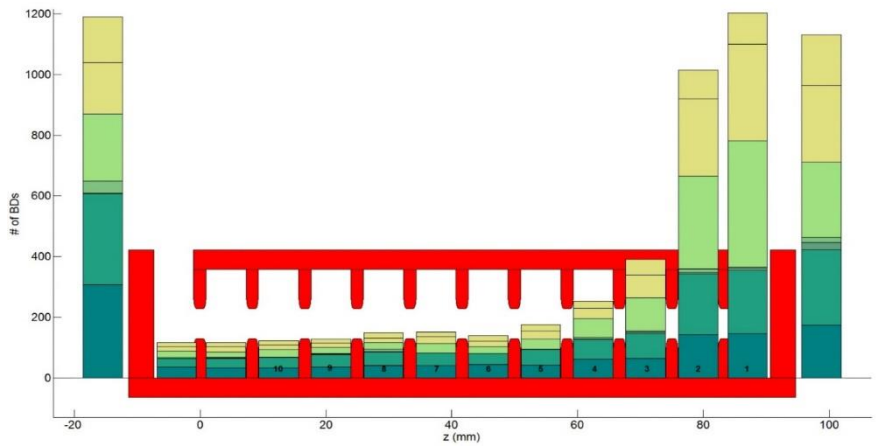
High Gradient testing



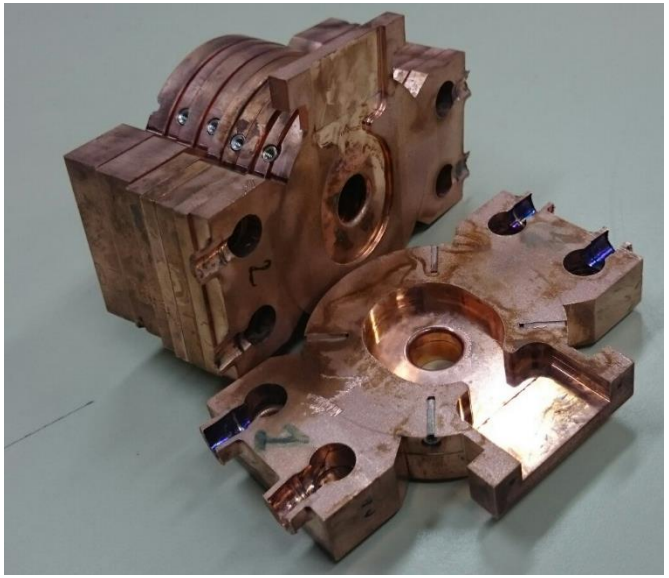
SEM
(Post-mortem)

Post-mortem: CLIC-AS

- Unfortunately not camera system on real structures
- Only solution to know the BD distribution, position and amount of affected surface

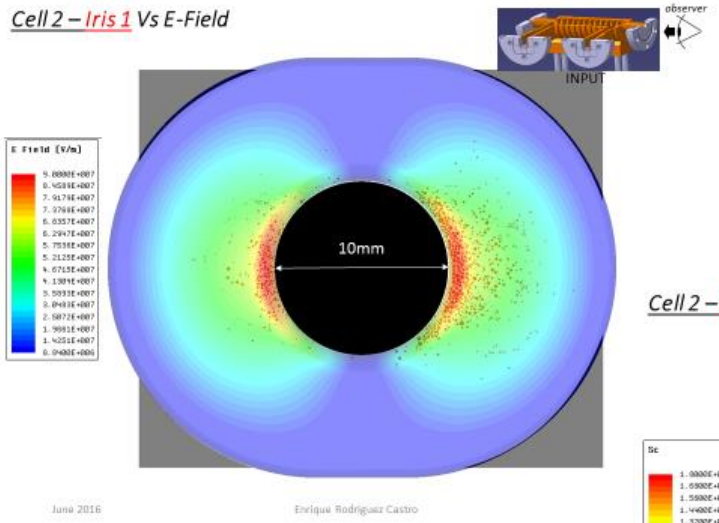


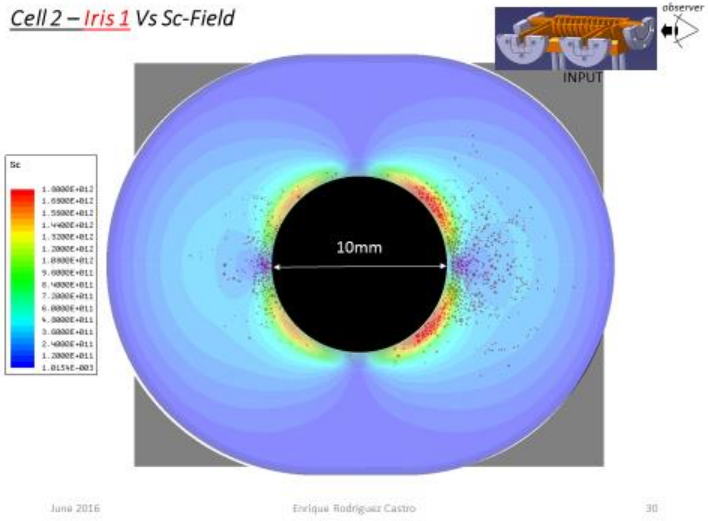
Histogram of the BD cell location, along with the month in which the BD occurred.

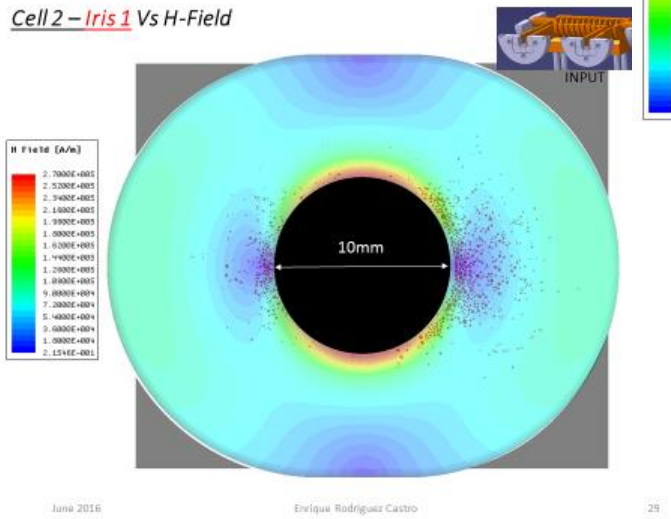


Crab cavity

- BD correspondence with the highest E-field on the cell

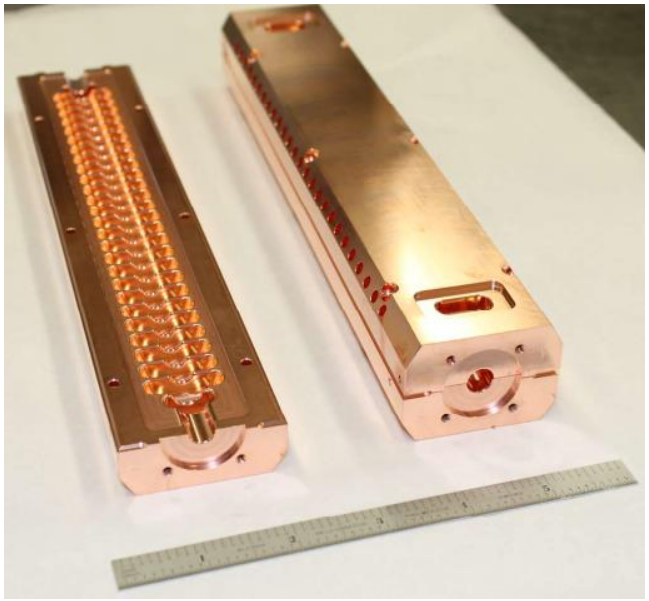


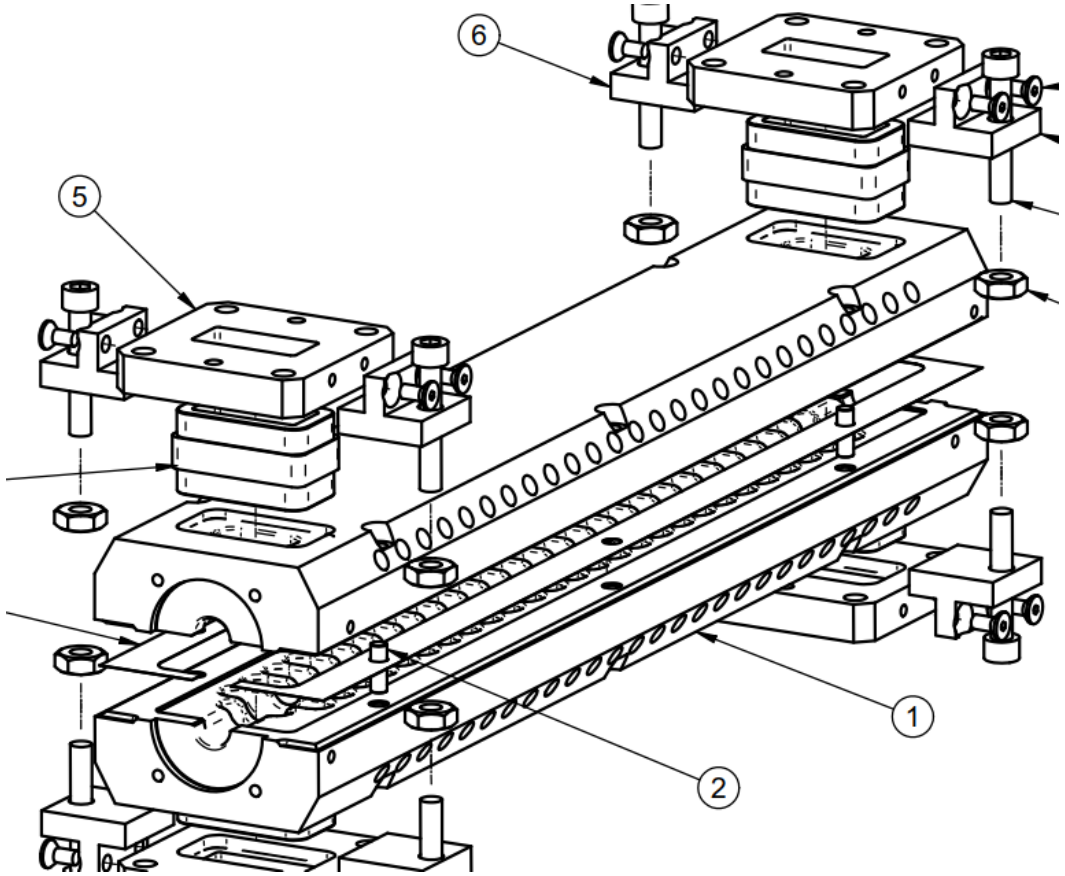




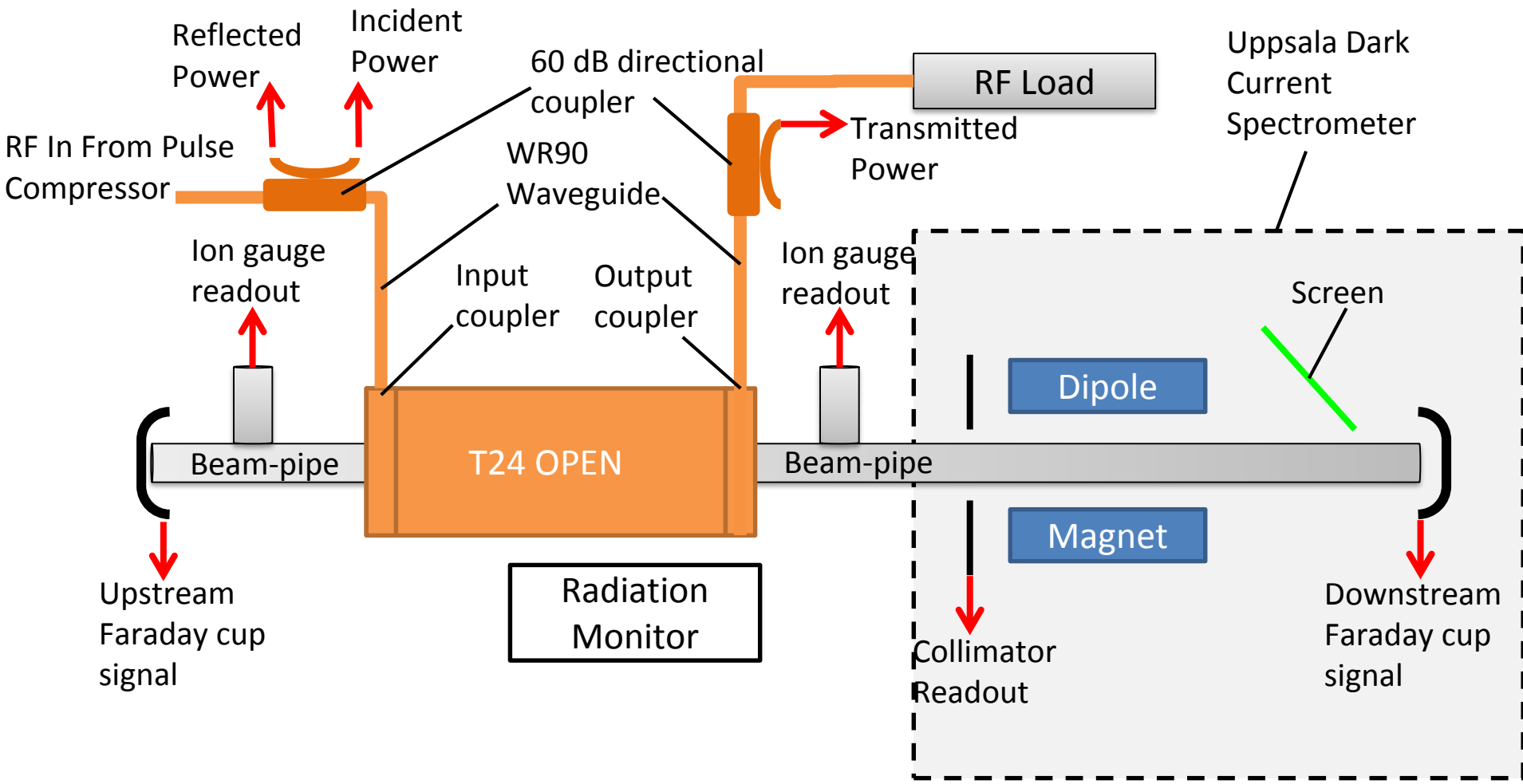
T24 Open

- Structure in halves
- Brazed using Au-Cu alloy as filler metal



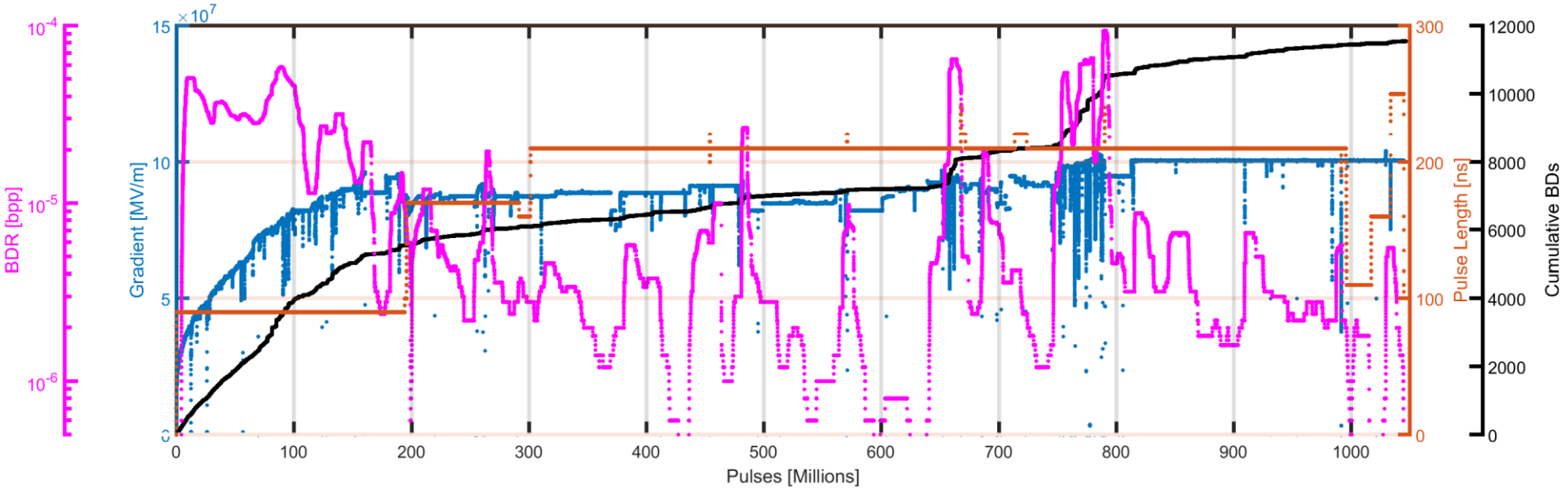


Xbox-2 Diagnostics



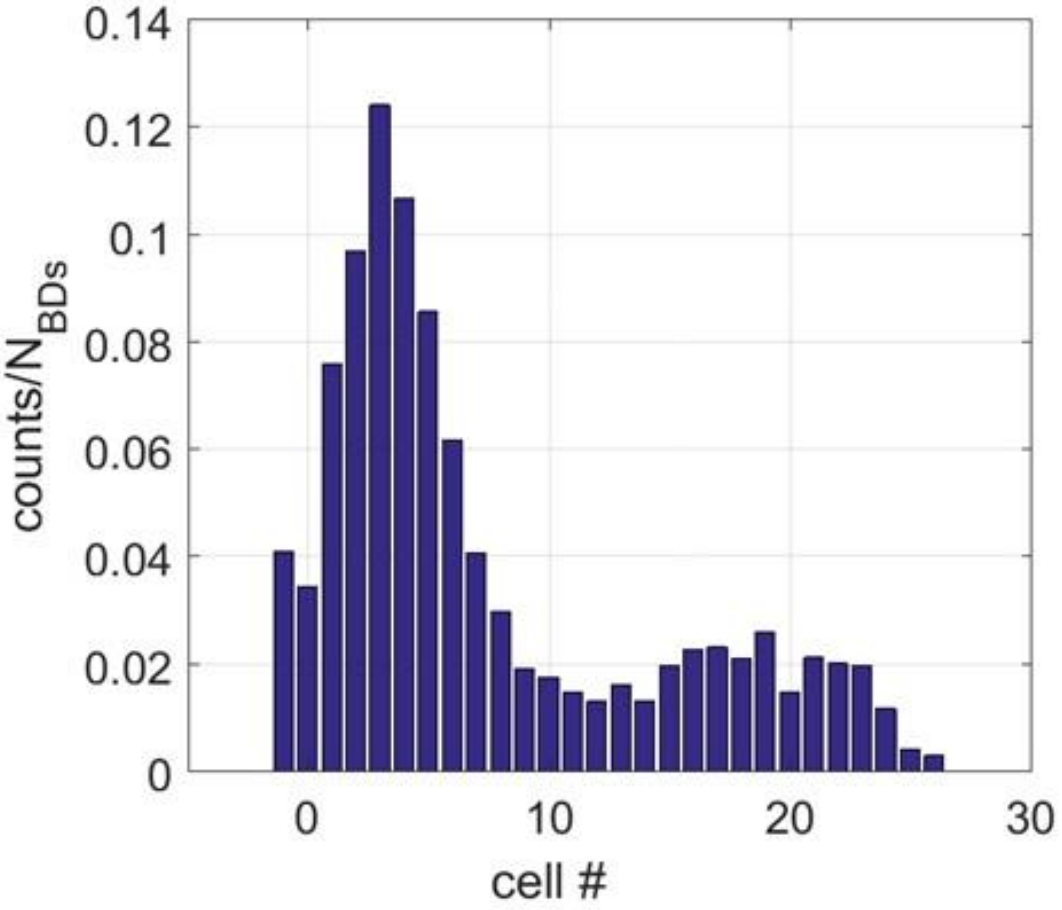
Tested at Xbox 2 from September 2015 to November 2016

Conditioning History



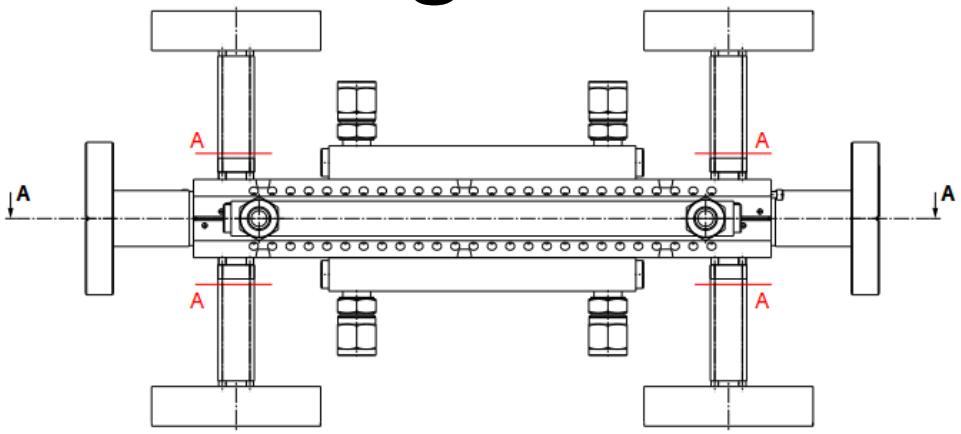
- Total number of pulses = 1.05×10^{9}
- Total number of BD = 11752
- Constant 3×10^{-5} bpp until reaching a gradient of 95 MV/m
  - Conditioning good but radiation issues on bunker thus gradient was reduced
  - Pulse length increased to 155 ns to increase power without interlocking the system.
- Finally 200ns pulse length for 100 MV/m with BDR of 10^{-6} bbp

Breakdown localization

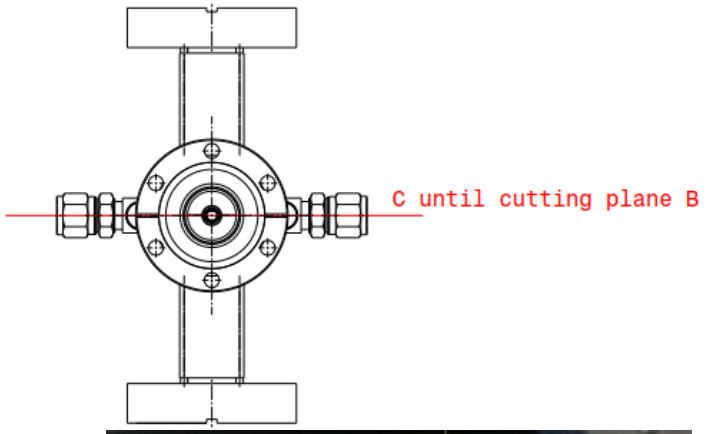


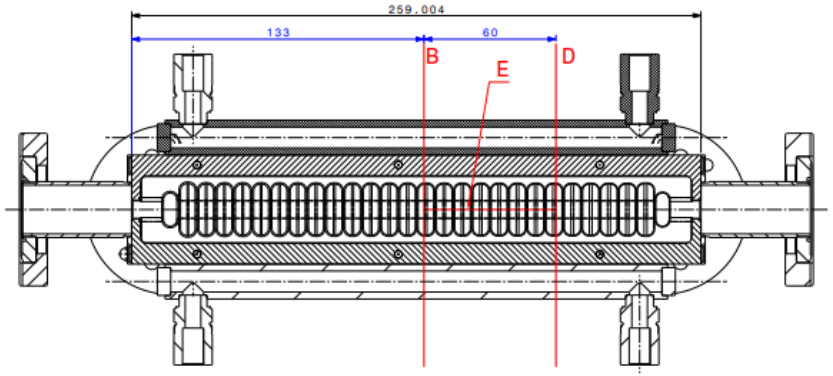
- Localisation of a breakdown was performed through a time-of-flight method.
- This method relies on the low group velocity of the structure, using the time of the rising edge of the reflected RF signal and falling transmitted signal.
- The difference between these signals will reflect the breakdown position

Cutting

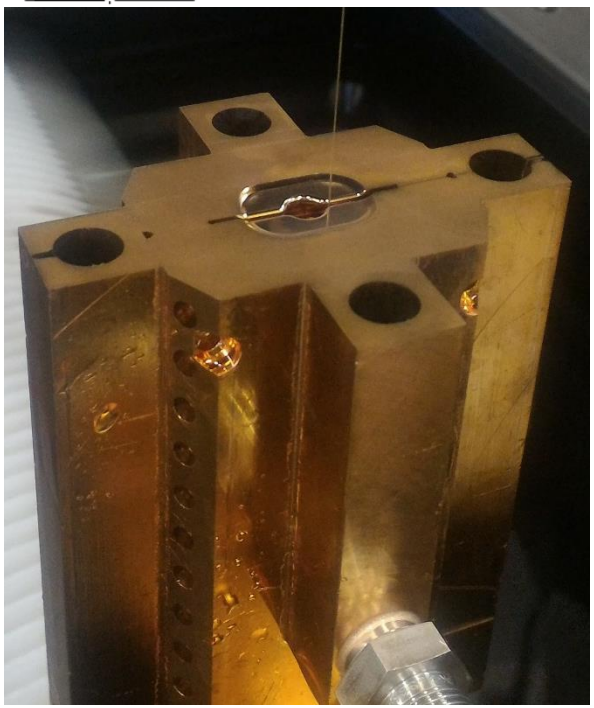


Front view
1:1



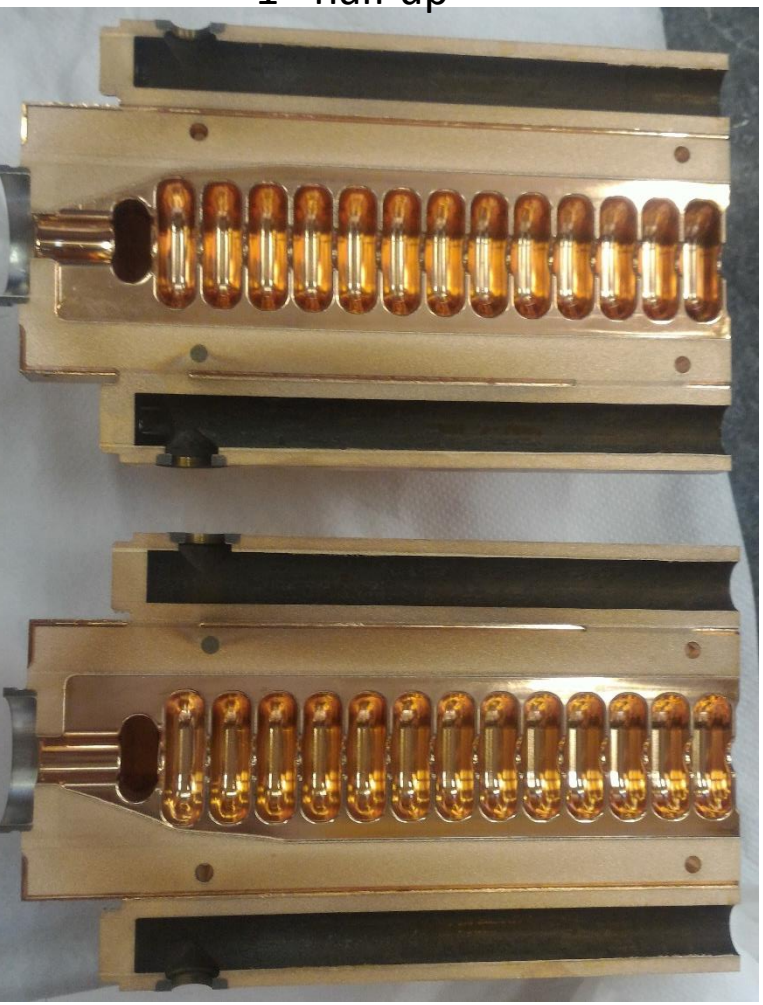


A-A
1:1



Nomenclature

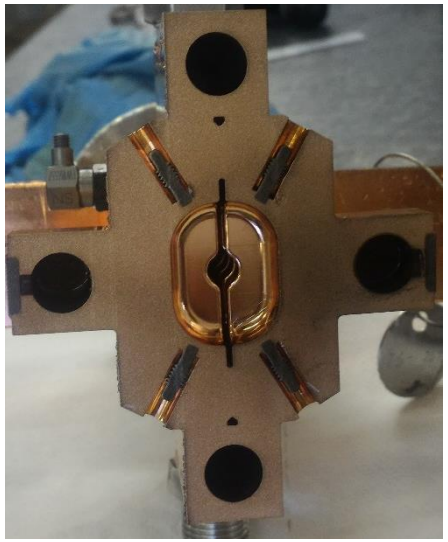
1st half up



1st half bottom

2nd cut left

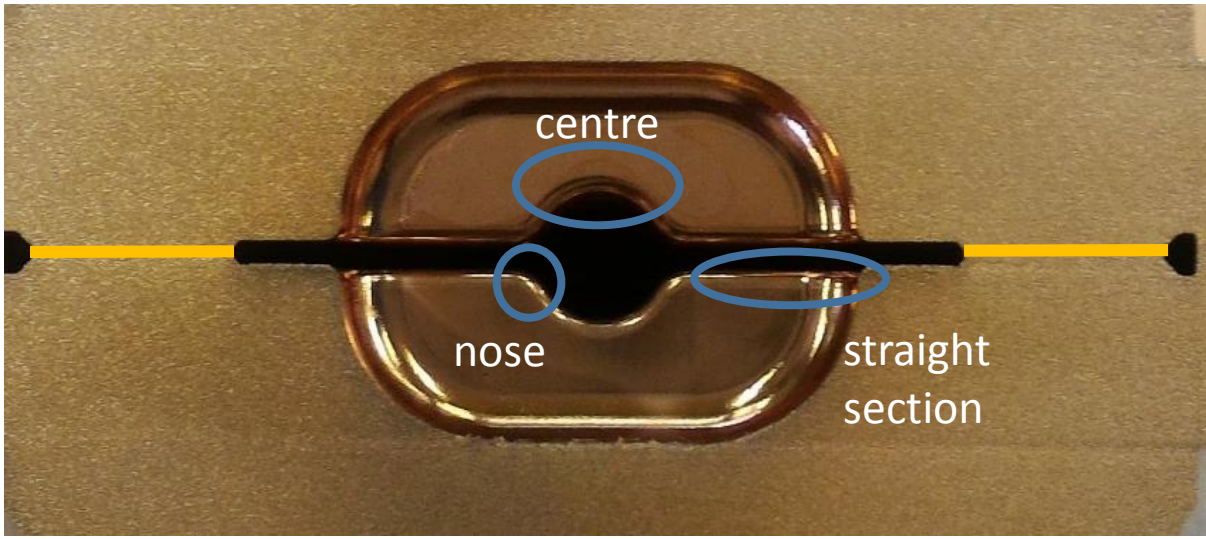
2nd cut right



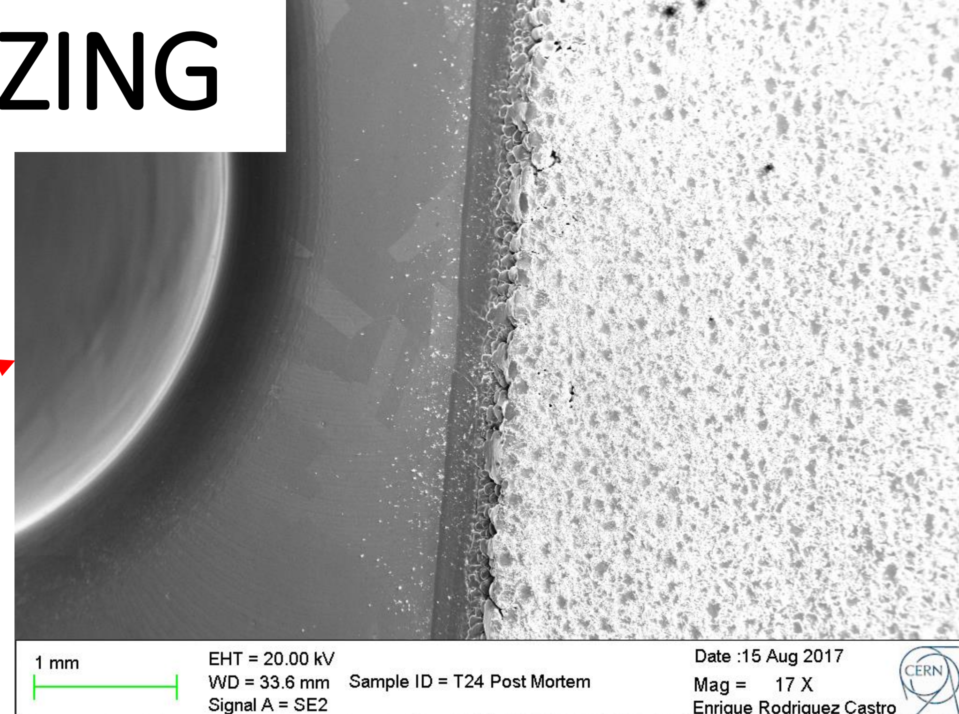
Flat view cell

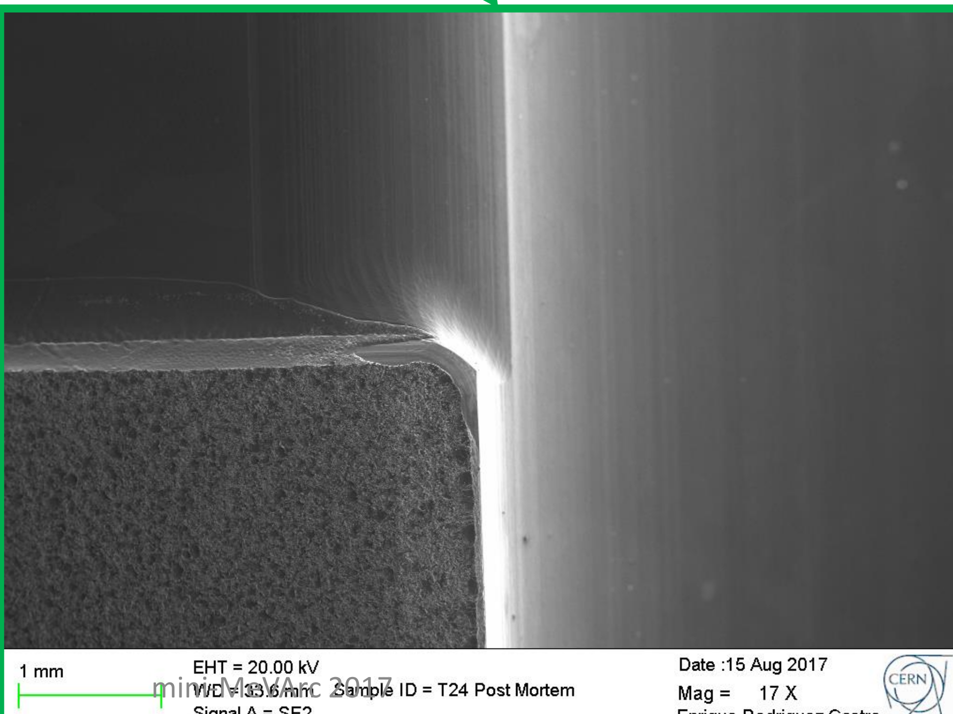
Nomenclature

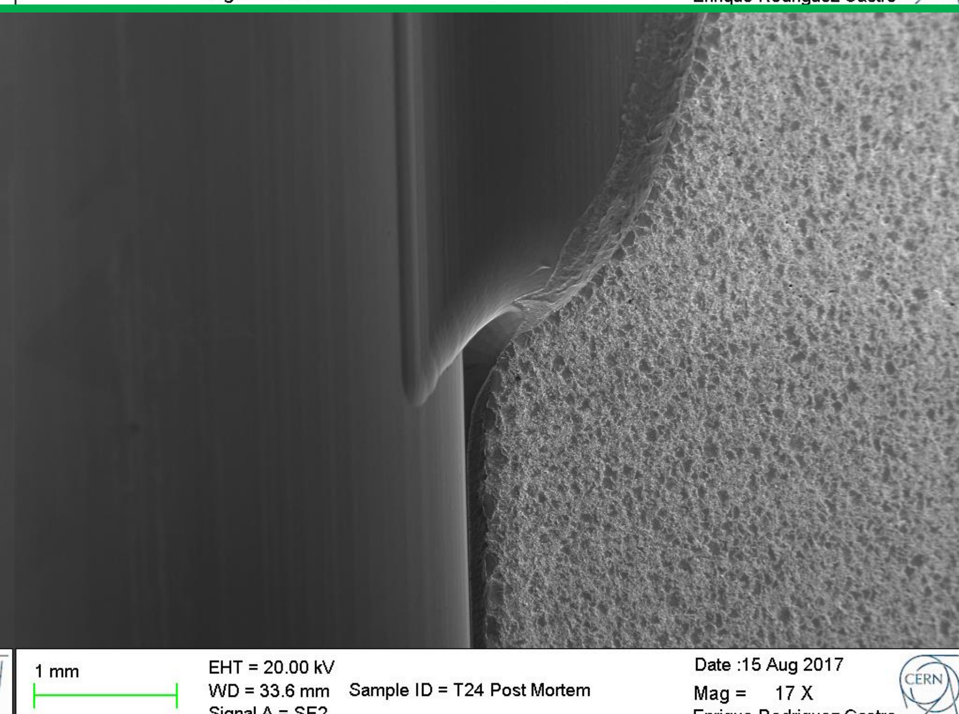
- Brazing → all features on the brazing or close to the brazing region related with BD activity
- Iris → here I'll make difference between 3 areas
  - Nose
  - Centre
  - Straight section

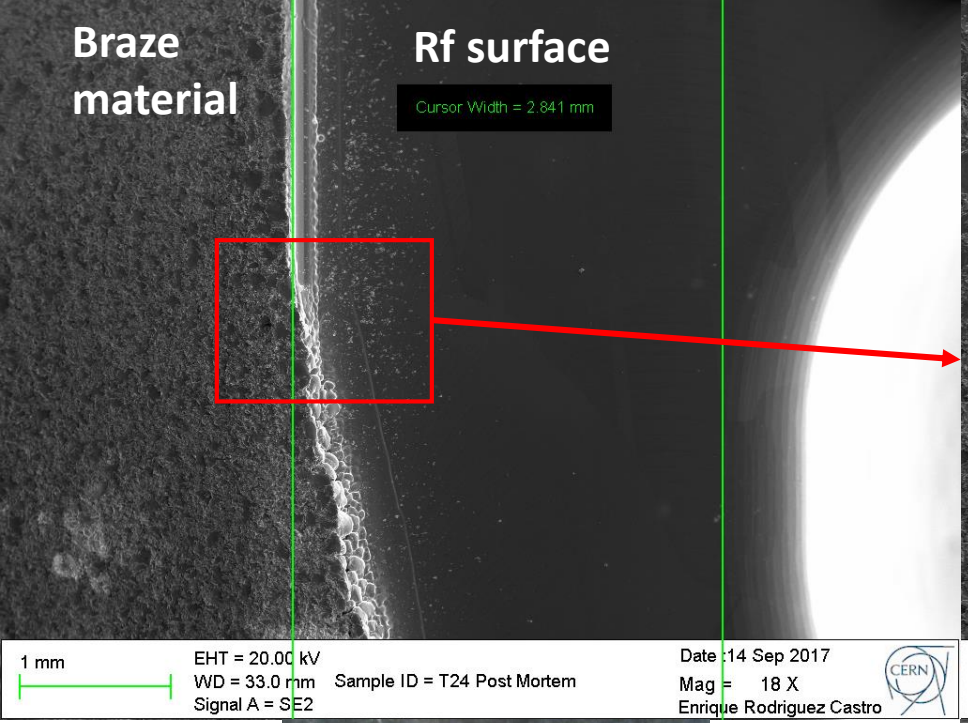


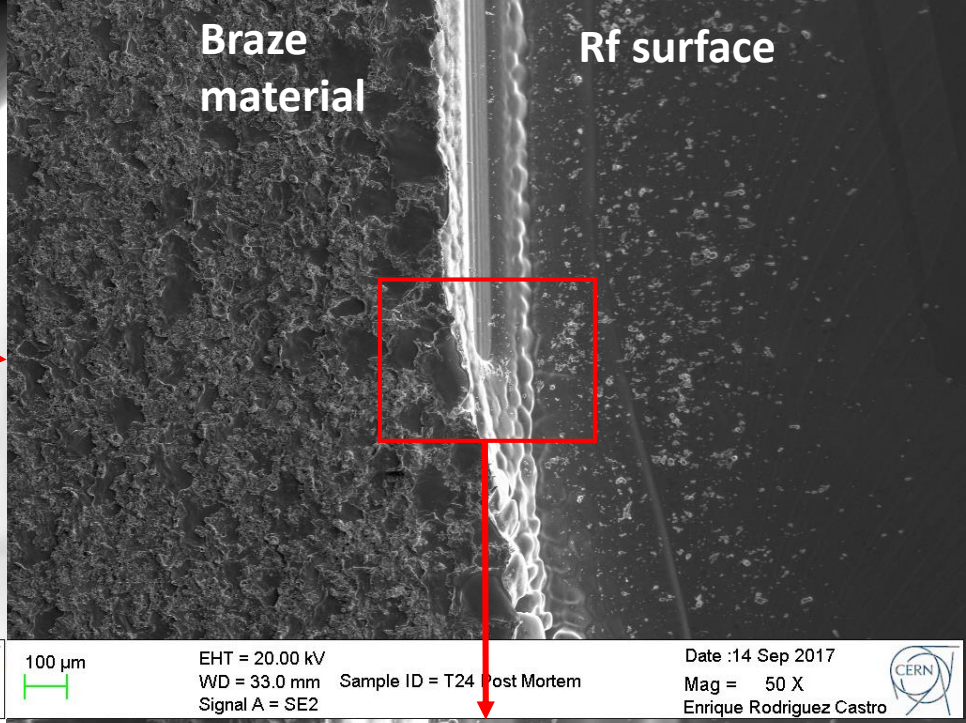
BRAZING

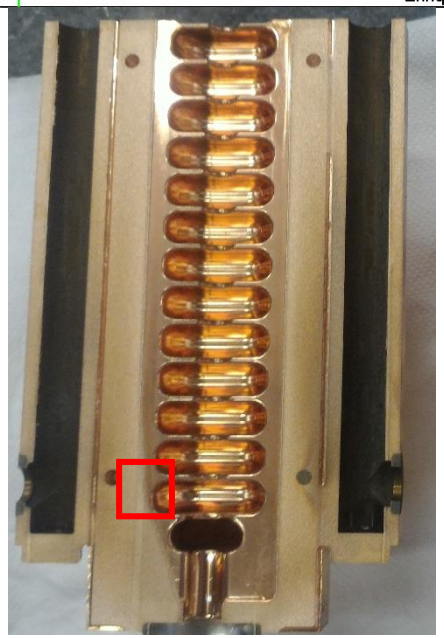






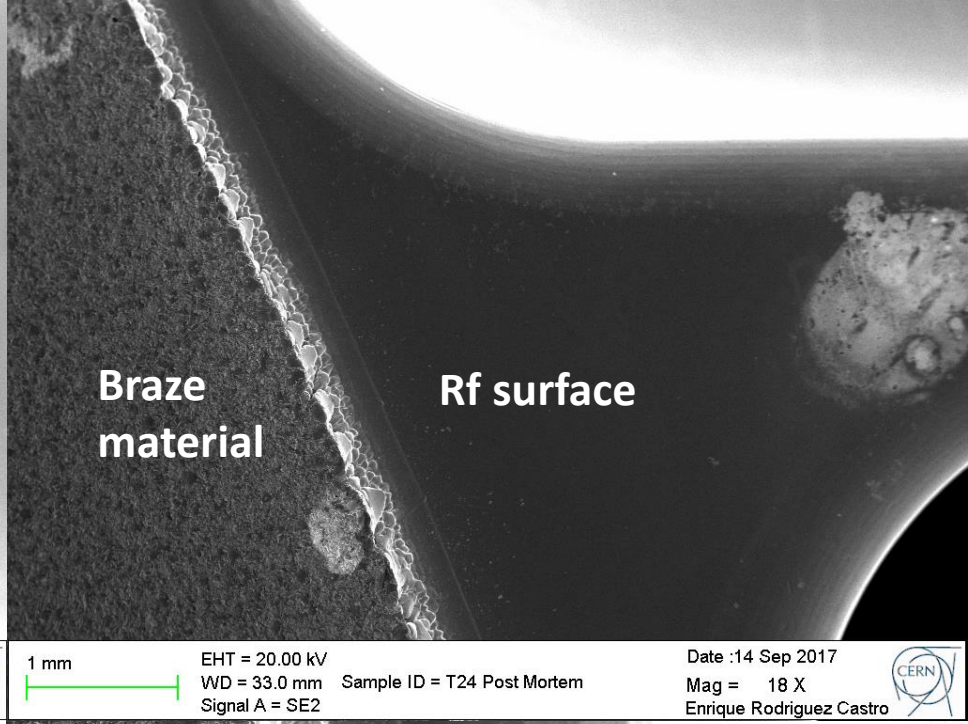

Braze material
Rf surface
Cursor Width = 2.841 mm
1 mm
EHT = 20.00 kV
WD = 33.0 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :14 Sep 2017
Mag = 18 X
Enrique Rodriguez Castro

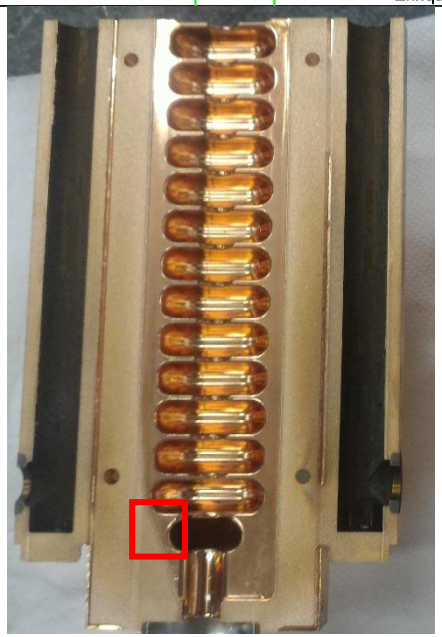

Braze material
Rf surface
100 µm
EHT = 20.00 kV
WD = 33.0 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :14 Sep 2017
Mag = 50 X
Enrique Rodriguez Castro

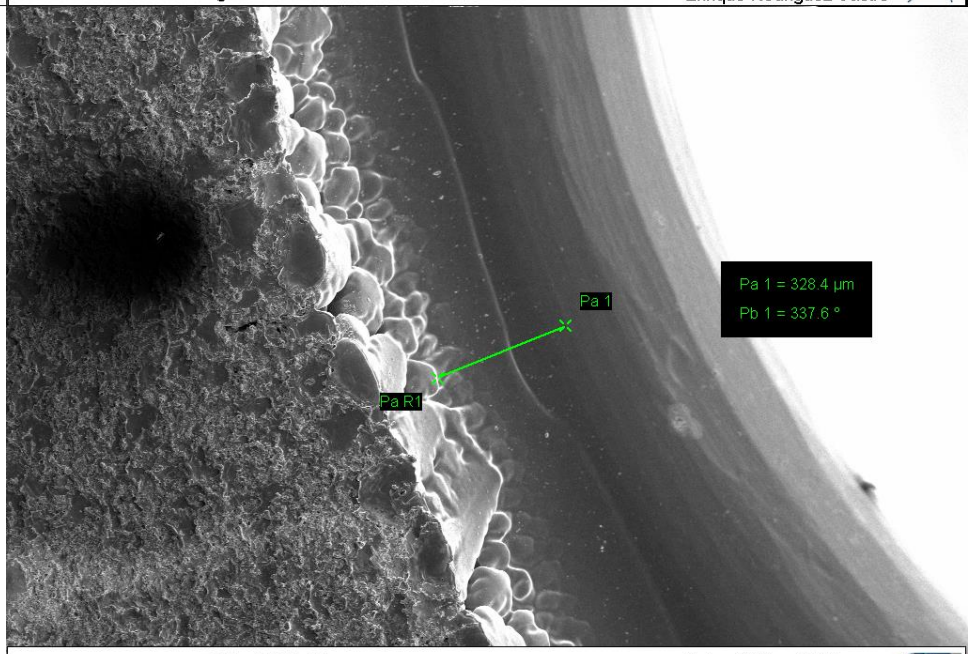


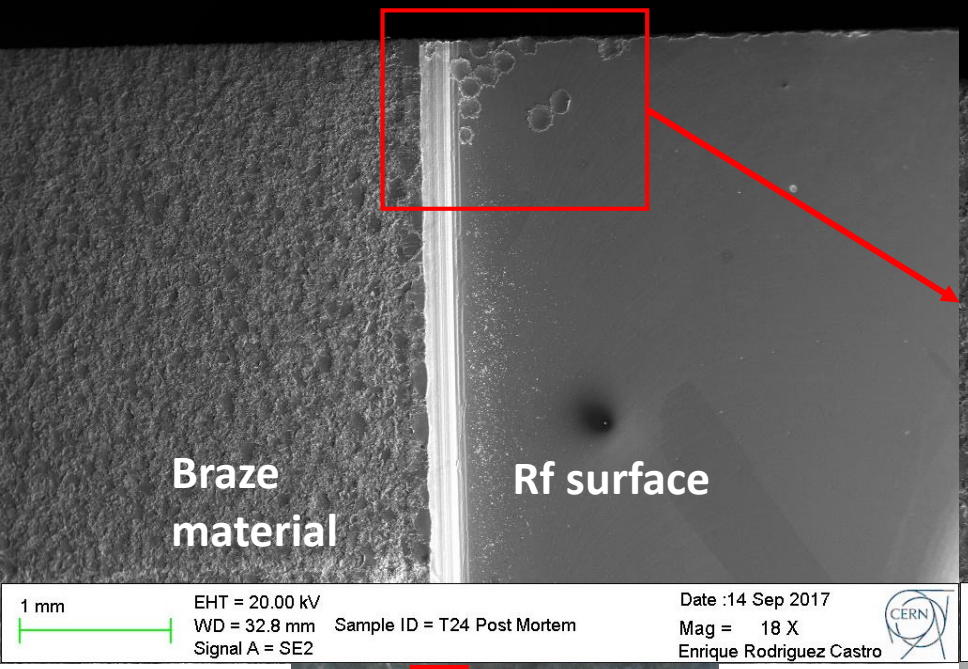

Braze material
Rf surface
20 µm
EHT = 20.00 kV
WD = 33.0 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :14 Sep 2017
Mag = 200 X
Enrique Rodriguez Castro

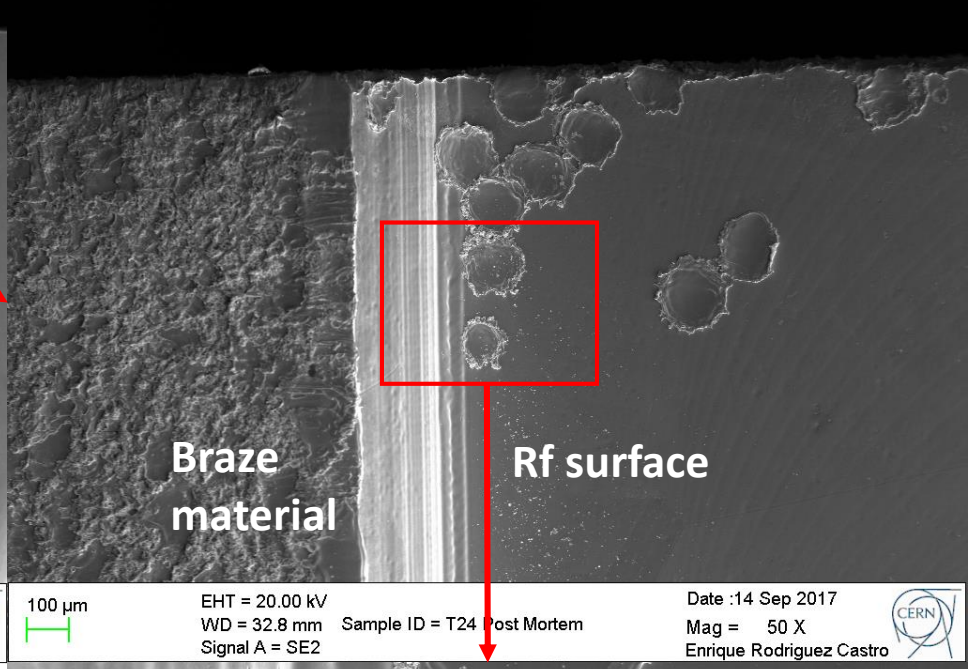

Braze material
Rf surface
Cursor Width = 521.0 µm
1 mm
EHT = 20.00 kV
WD = 33.0 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :14 Sep 2017
Mag = 18 X
Enrique Rodriguez Castro

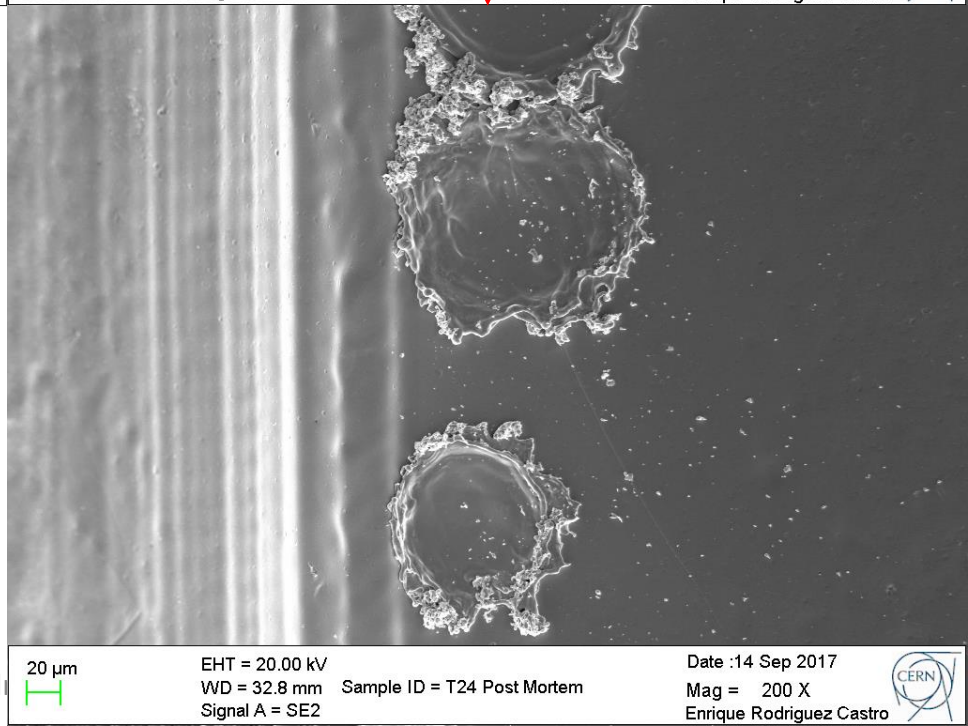

Braze material
Rf surface
1 mm
EHT = 20.00 kV
WD = 33.0 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :14 Sep 2017
Mag = 18 X
Enrique Rodriguez Castro

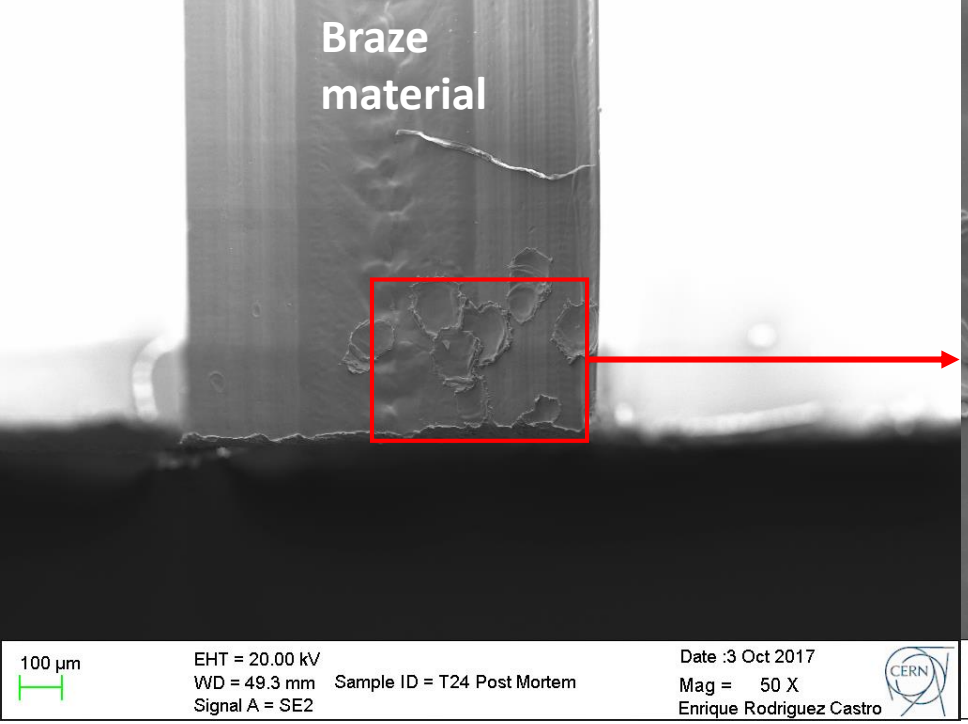


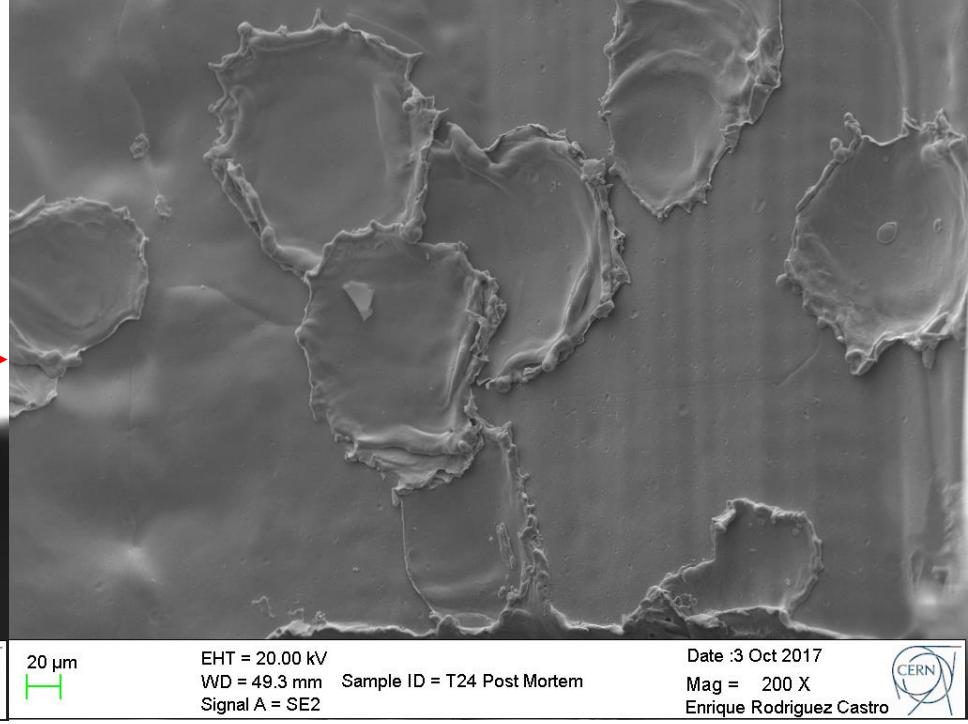

Pa 1
Pa R1
Pa 1 = 328.4 µm
Pb 1 = 337.6 °
100 µm
EHT = 20.00 kV
WD = 33.0 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :14 Sep 2017
Mag = 50 X
Enrique Rodriguez Castro

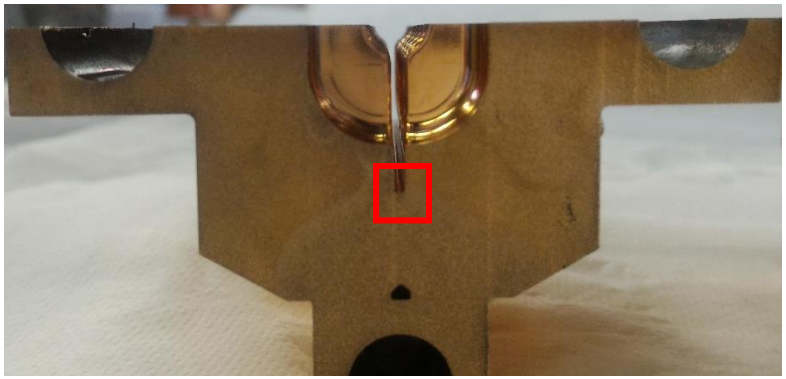

Braze material
Rf surface
1 mm
EHT = 20.00 kV
WD = 32.8 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :14 Sep 2017
Mag = 18 X
Enrique Rodriguez Castro

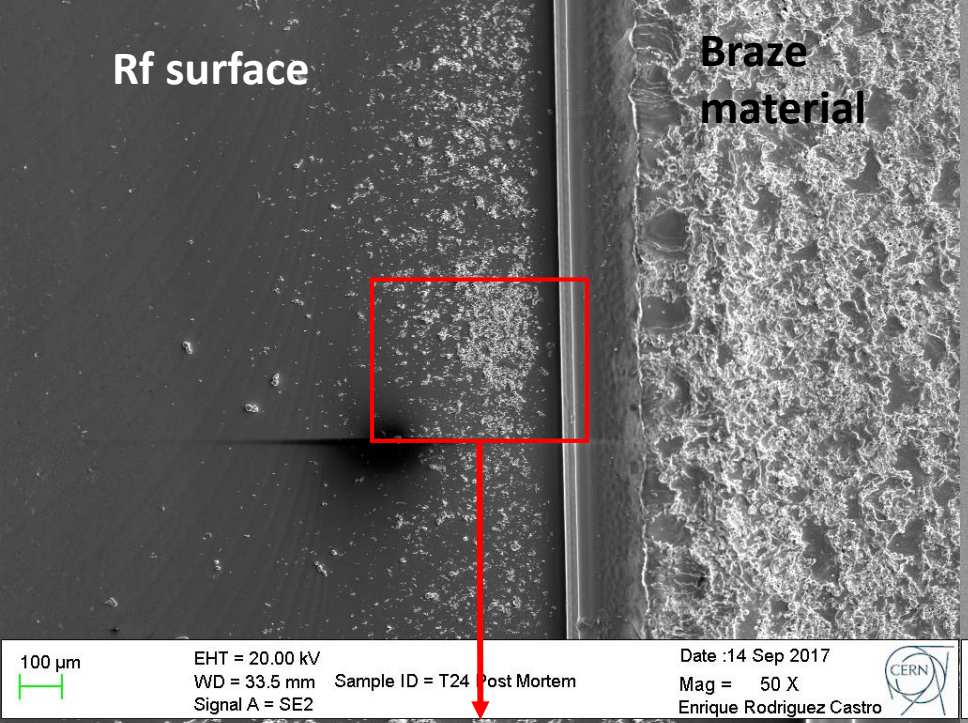

Braze material
Rf surface
100 µm
EHT = 20.00 kV
WD = 32.8 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :14 Sep 2017
Mag = 50 X
Enrique Rodriguez Castro

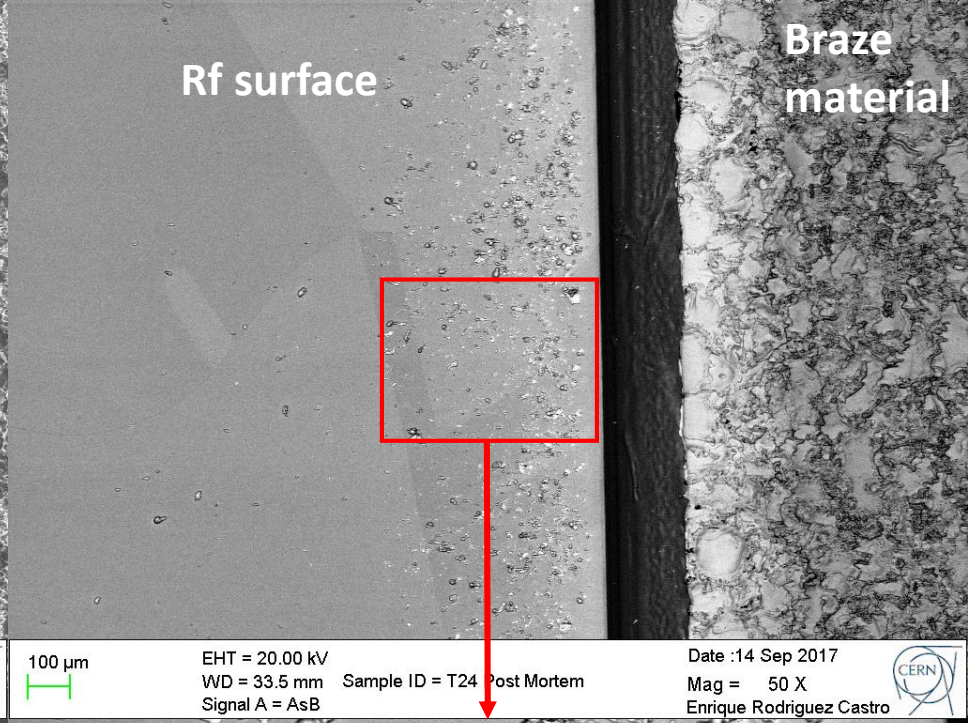

20 µm
EHT = 20.00 kV
WD = 32.8 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :14 Sep 2017
Mag = 200 X
Enrique Rodriguez Castro

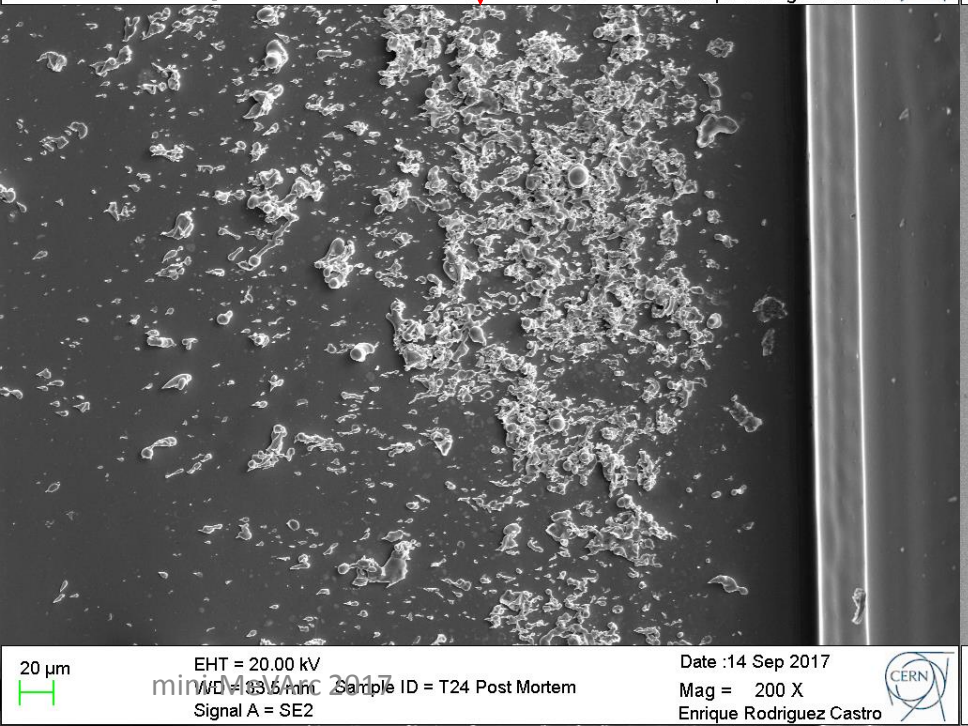

Braze
material
100 µm
EHT = 20.00 kV
WD = 49.3 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :3 Oct 2017
Mag = 50 X
Enrique Rodriguez Castro
CERN

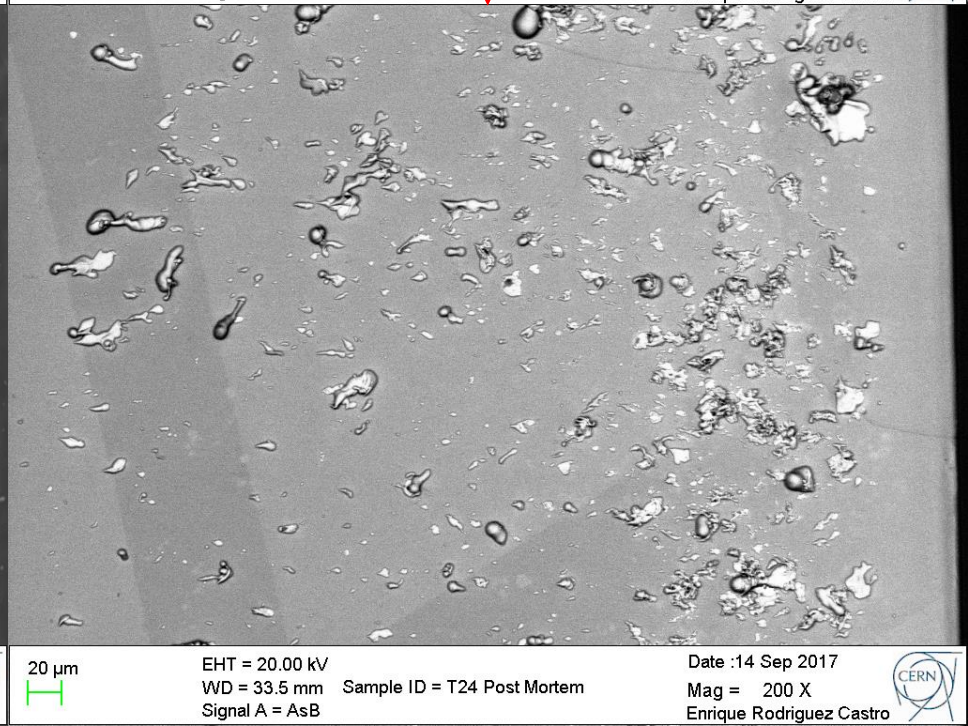

20 µm
EHT = 20.00 kV
WD = 49.3 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :3 Oct 2017
Mag = 200 X
Enrique Rodriguez Castro
CERN





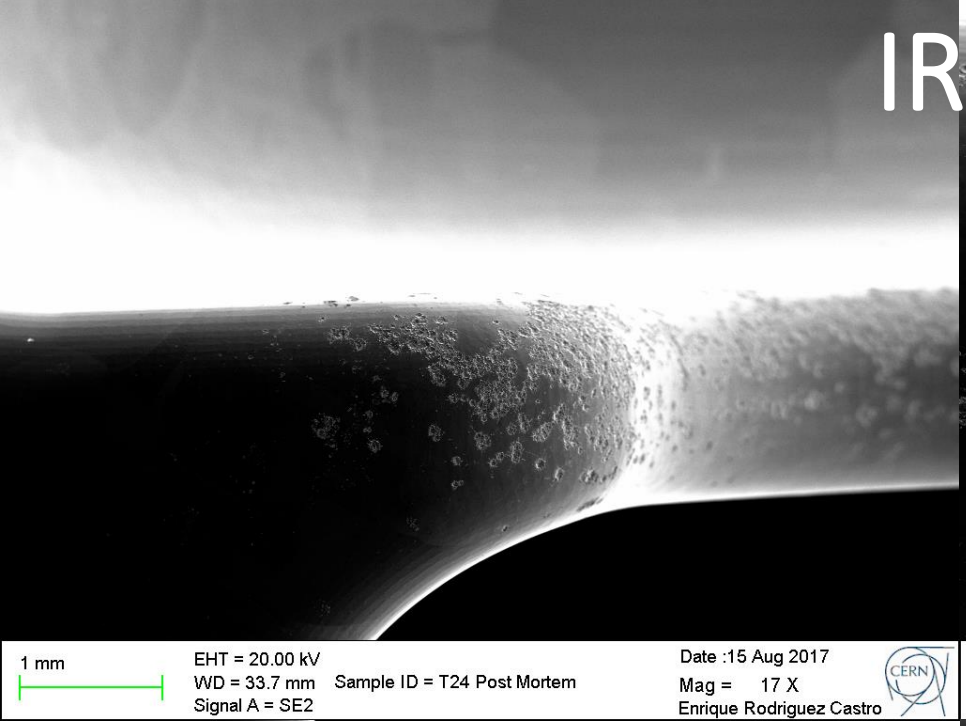


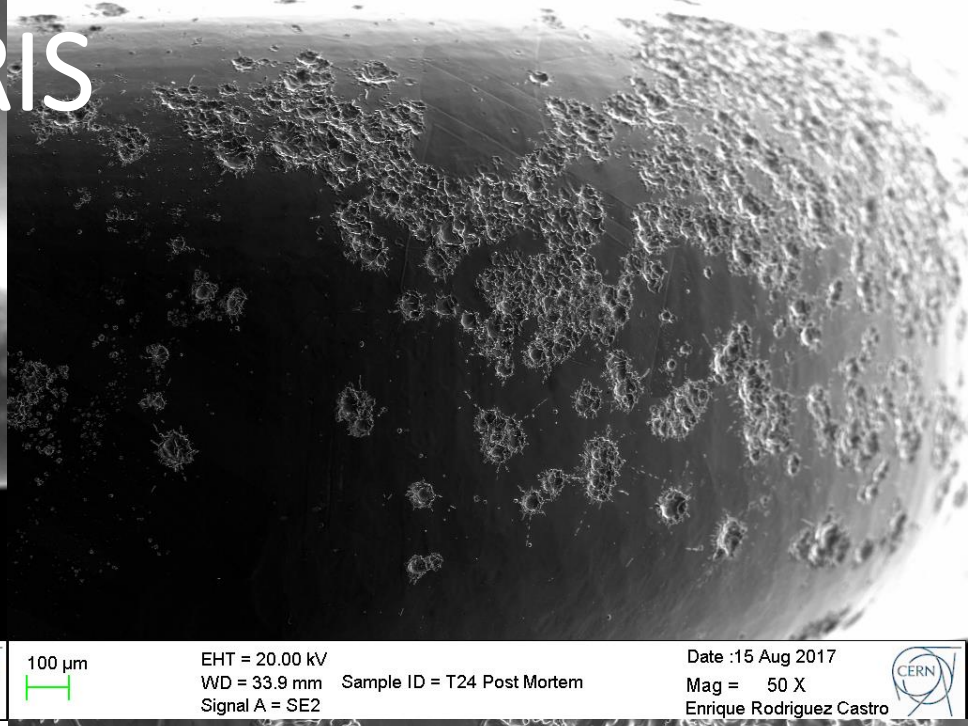


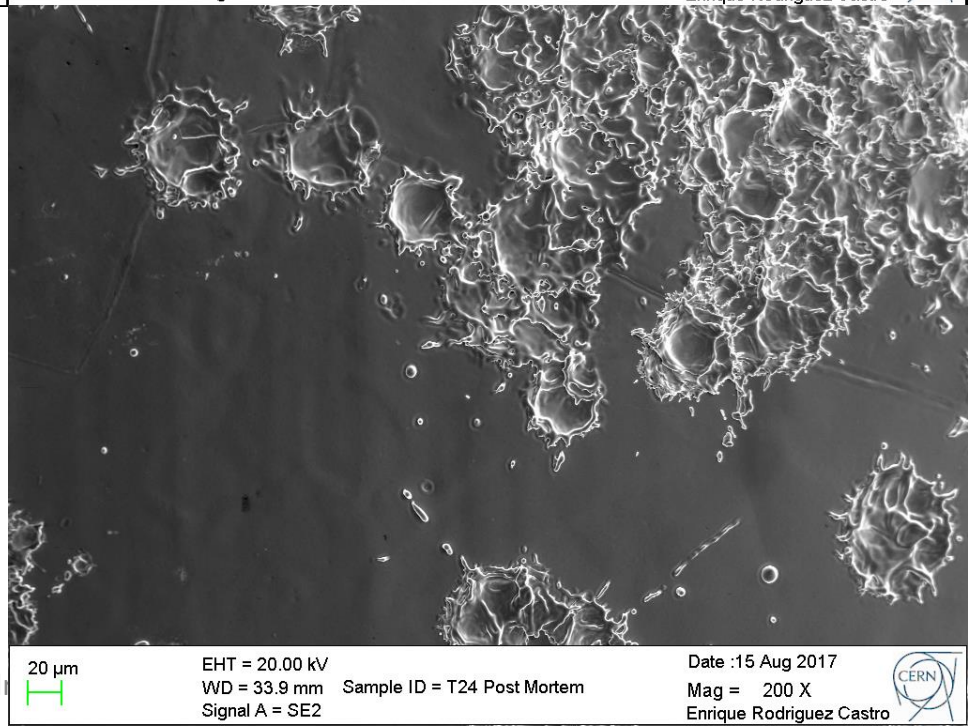


IRIS

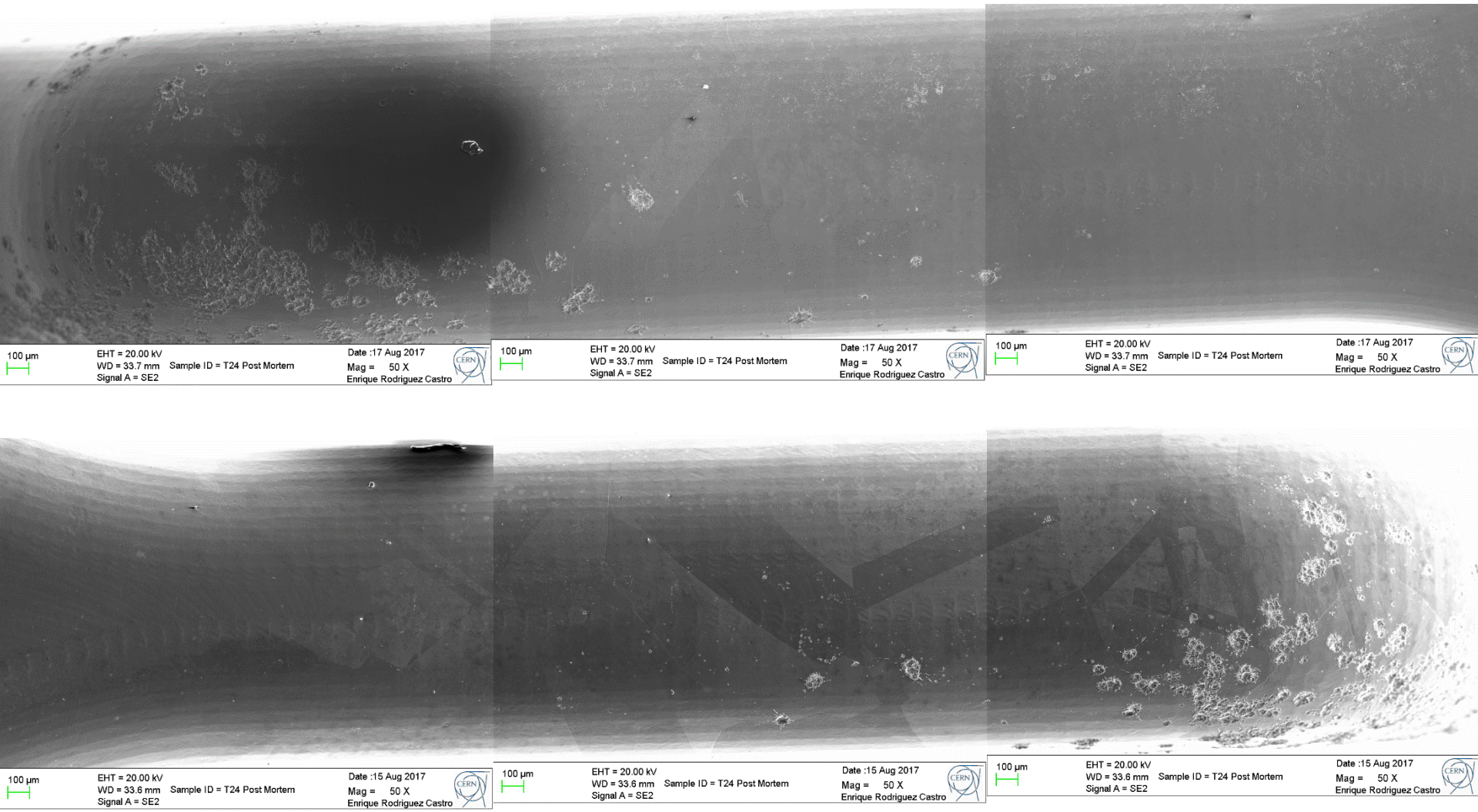
IRIS

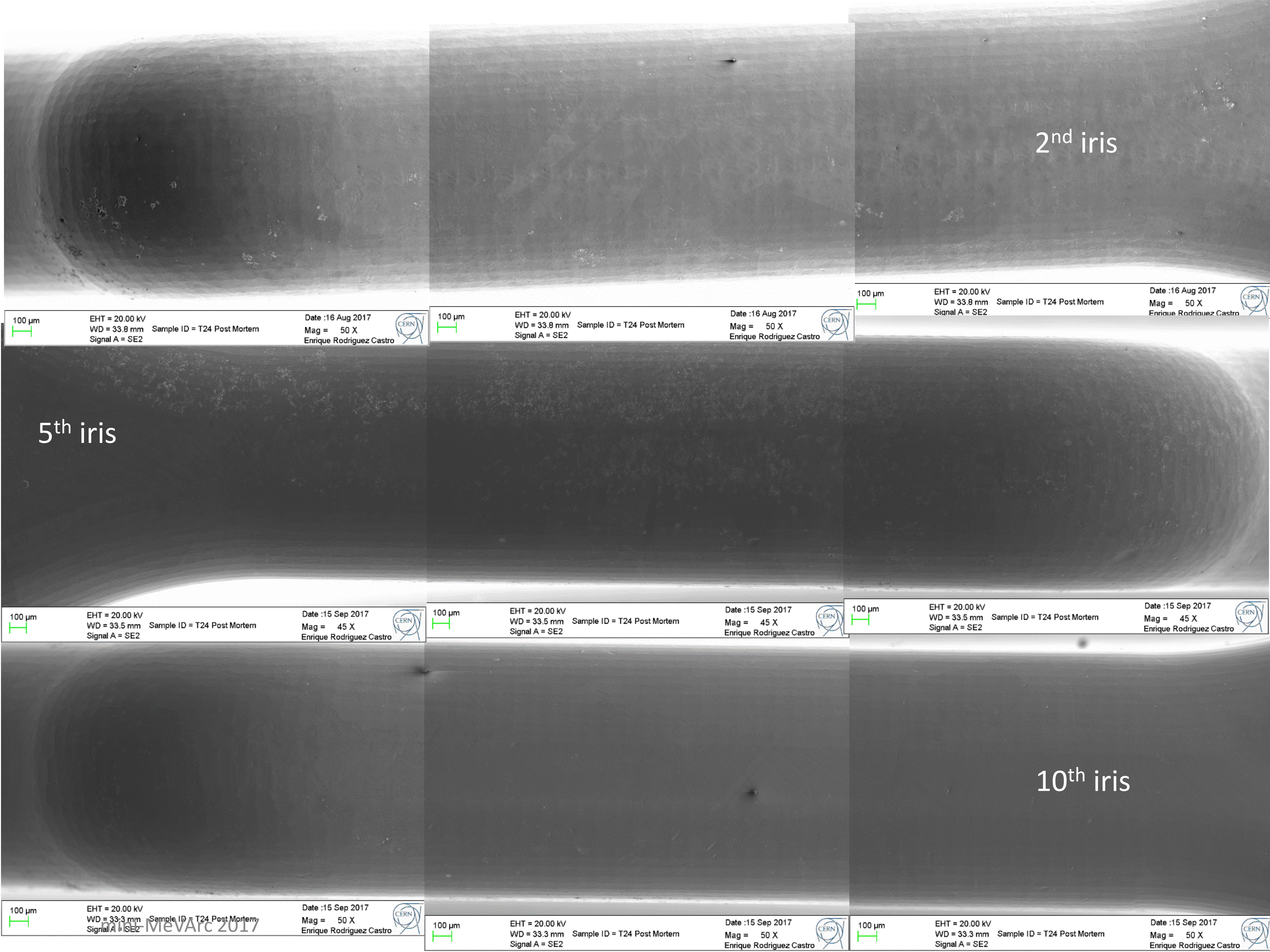

1 mm
EHT = 20.00 kV
WD = 33.7 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :15 Aug 2017
Mag = 17 X
Enrique Rodriguez Castro


100 µm
EHT = 20.00 kV
WD = 33.9 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :15 Aug 2017
Mag = 50 X
Enrique Rodriguez Castro

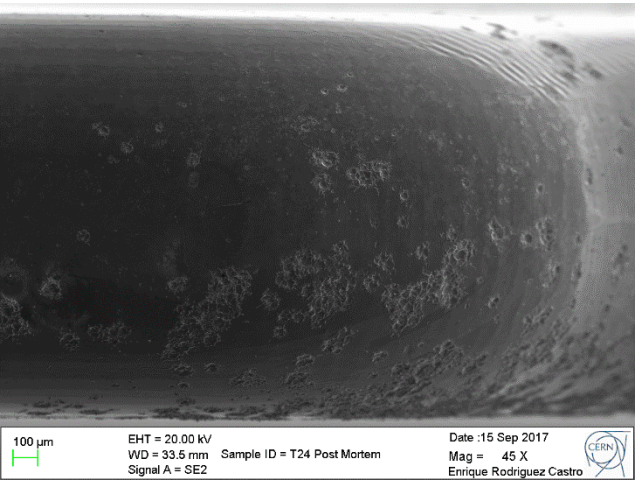

20 µm
EHT = 20.00 kV
WD = 33.9 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :15 Aug 2017
Mag = 200 X
Enrique Rodriguez Castro

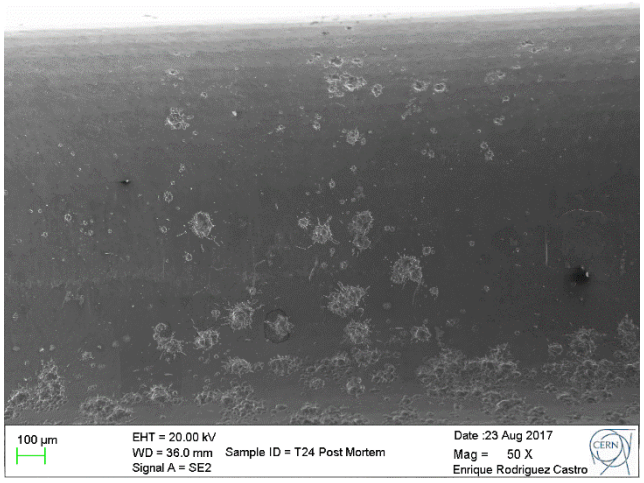
First iris

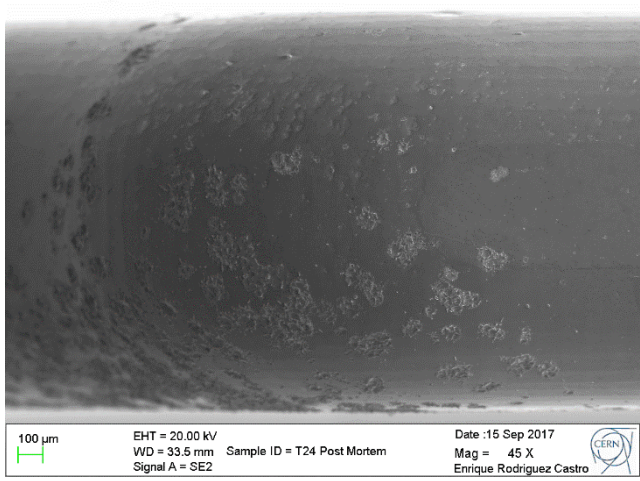

100 µm
EHT = 20.00 kV
WD = 33.7 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :17 Aug 2017
Mag = 50 X
Enrique Rodriguez Castro
100 µm
EHT = 20.00 kV
WD = 33.7 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :17 Aug 2017
Mag = 50 X
Enrique Rodriguez Castro
100 µm
EHT = 20.00 kV
WD = 33.7 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :17 Aug 2017
Mag = 50 X
Enrique Rodriguez Castro
100 µm
EHT = 20.00 kV
WD = 33.6 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :15 Aug 2017
Mag = 50 X
Enrique Rodriguez Castro
100 µm
EHT = 20.00 kV
WD = 33.6 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :15 Aug 2017
Mag = 50 X
Enrique Rodriguez Castro
100 µm
EHT = 20.00 kV
WD = 33.6 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :15 Aug 2017
Mag = 50 X
Enrique Rodriguez Castro



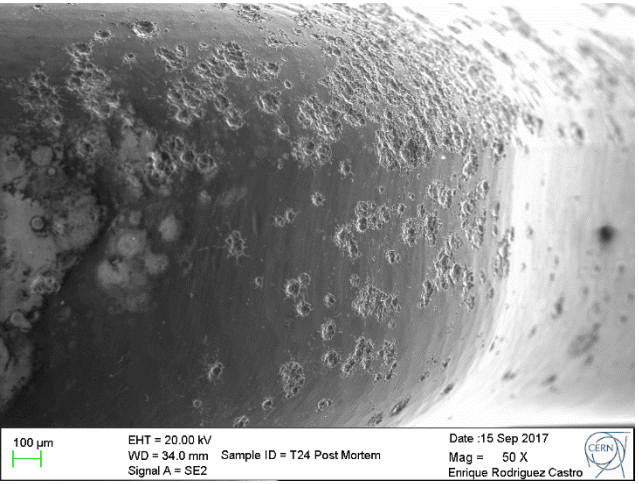
2nd iris
5th iris
10th iris
100 µm
EHT = 20.00 kV
WD = 33.8 mm
Sample ID = T24 Post Mortem
Signal A = SE2
Date :16 Aug 2017
Mag = 50 X
Enrique Rodriguez Castro
CERN
100 µm
EHT = 20.00 kV
WD = 33.8 mm
Sample ID = T24 Post Mortem
Signal A = SE2
Date :16 Aug 2017
Mag = 50 X
Enrique Rodriguez Castro
CERN
100 µm
EHT = 20.00 kV
WD = 33.8 mm
Sample ID = T24 Post Mortem
Signal A = SE2
Date :16 Aug 2017
Mag = 50 X
Enrique Rodriguez Castro
CERN
100 µm
EHT = 20.00 kV
WD = 33.5 mm
Sample ID = T24 Post Mortem
Signal A = SE2
Date :15 Sep 2017
Mag = 45 X
Enrique Rodriguez Castro
CERN
100 µm
EHT = 20.00 kV
WD = 33.5 mm
Sample ID = T24 Post Mortem
Signal A = SE2
Date :15 Sep 2017
Mag = 45 X
Enrique Rodriguez Castro
CERN
100 µm
EHT = 20.00 kV
WD = 33.5 mm
Sample ID = T24 Post Mortem
Signal A = SE2
Date :15 Sep 2017
Mag = 45 X
Enrique Rodriguez Castro
CERN
100 µm
EHT = 20.00 kV
WD = 33.3 mm
Sample ID = T24 Post Mortem
Signal A = SE2
Date :15 Sep 2017
Mag = 50 X
Enrique Rodriguez Castro
CERN
100 µm
EHT = 20.00 kV
WD = 33.3 mm
Sample ID = T24 Post Mortem
Signal A = SE2
Date :15 Sep 2017
Mag = 50 X
Enrique Rodriguez Castro
CERN
100 µm
EHT = 20.00 kV
WD = 33.3 mm
Sample ID = T24 Post Mortem
Signal A = SE2
Date :15 Sep 2017
Mag = 50 X
Enrique Rodriguez Castro
CERN

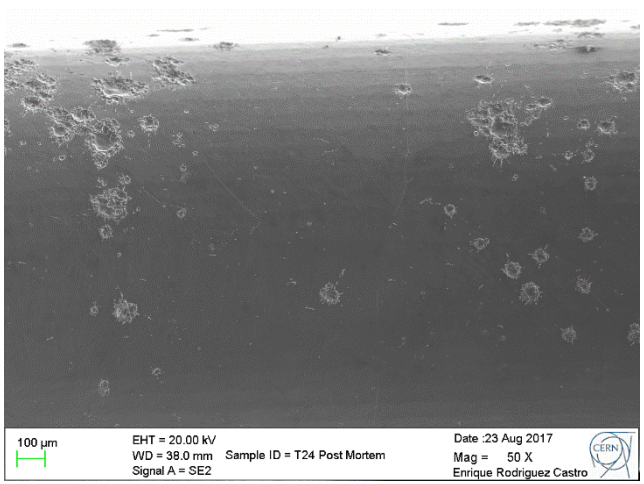


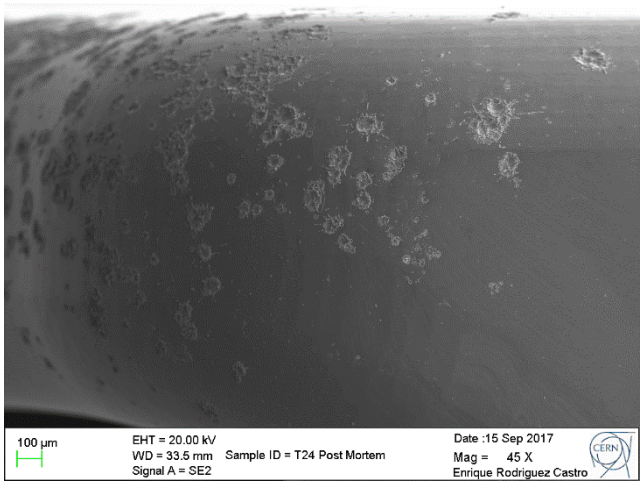




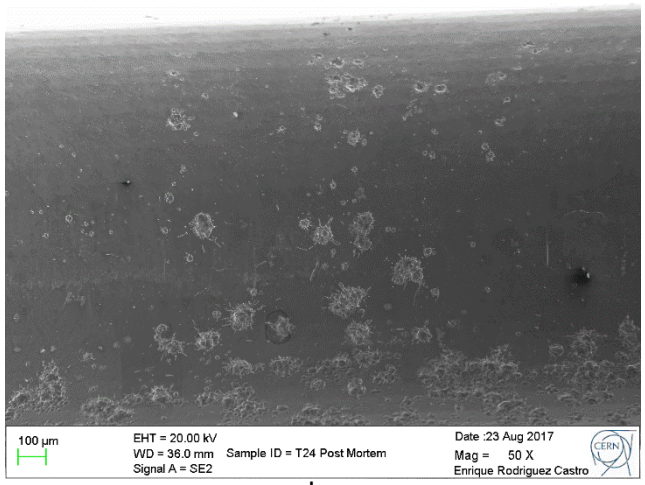
2nd iris

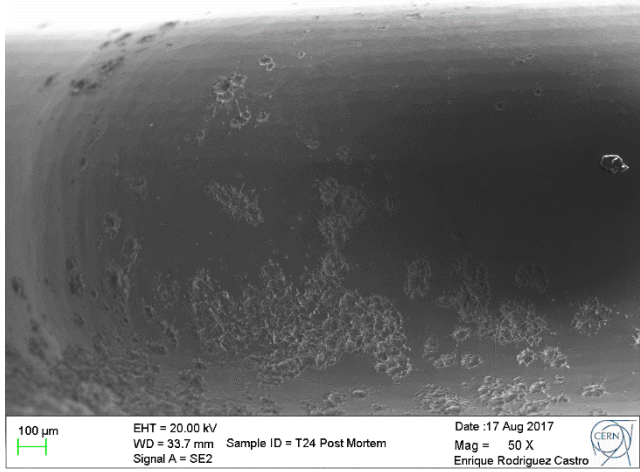




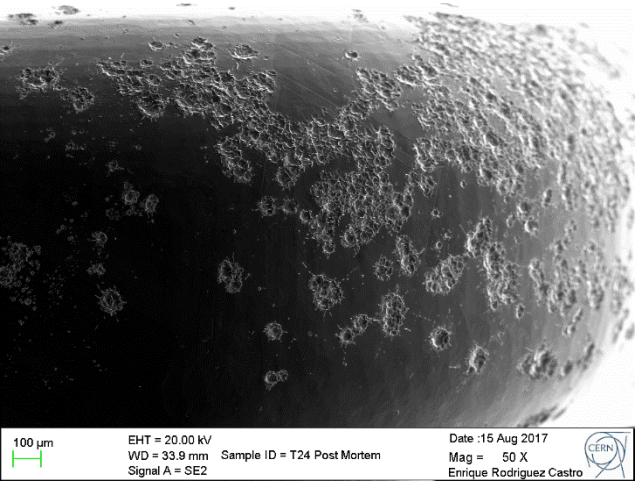


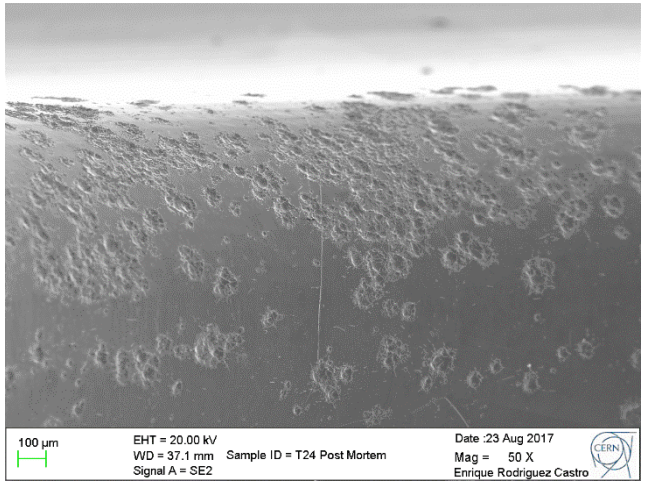
1st iris

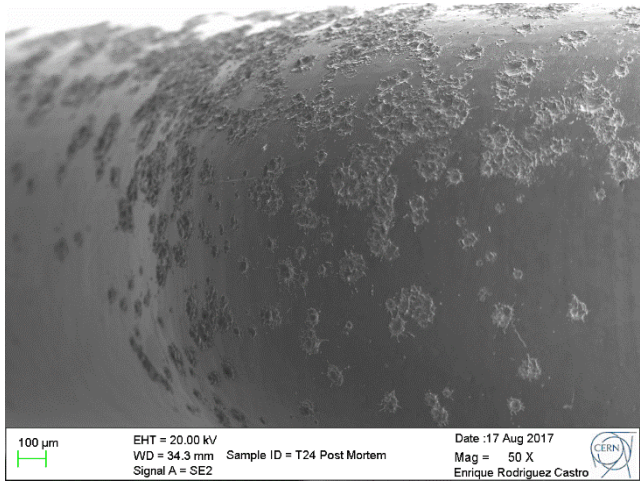




2nd iris

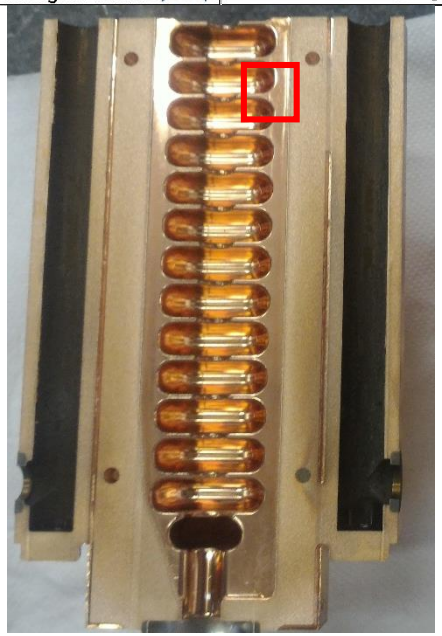


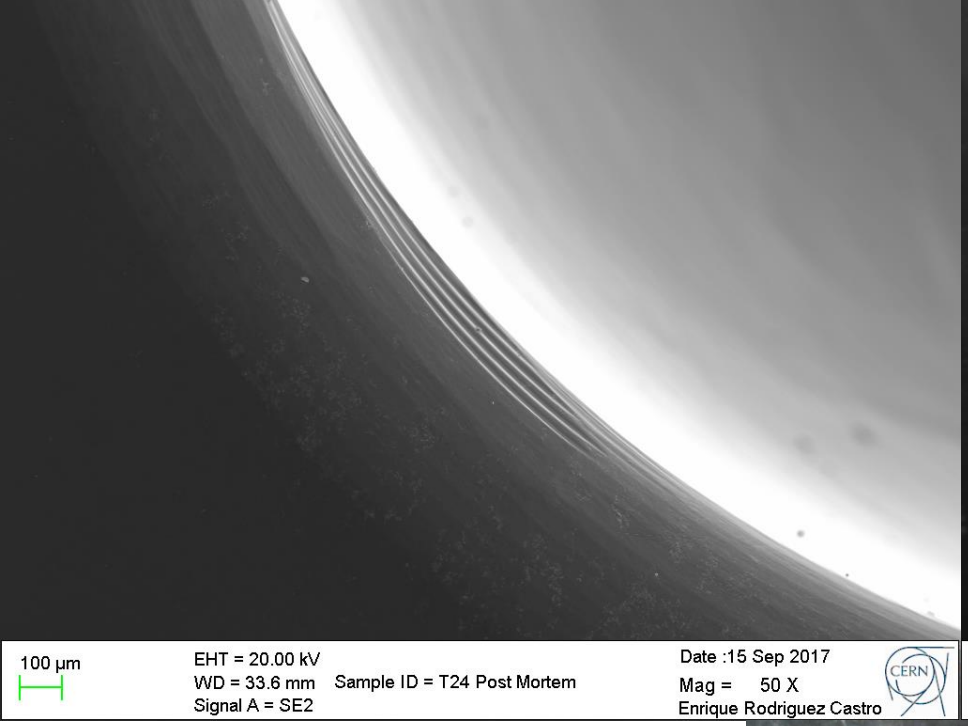


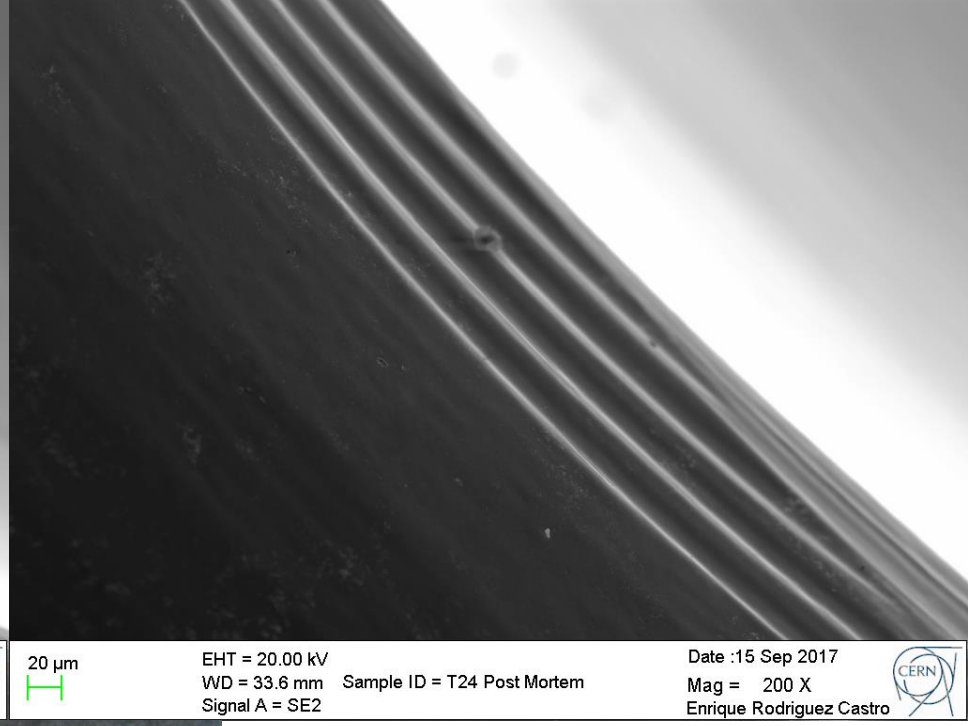


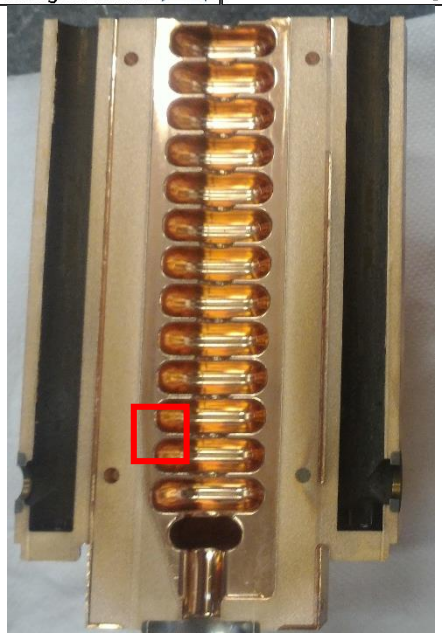
1st iris

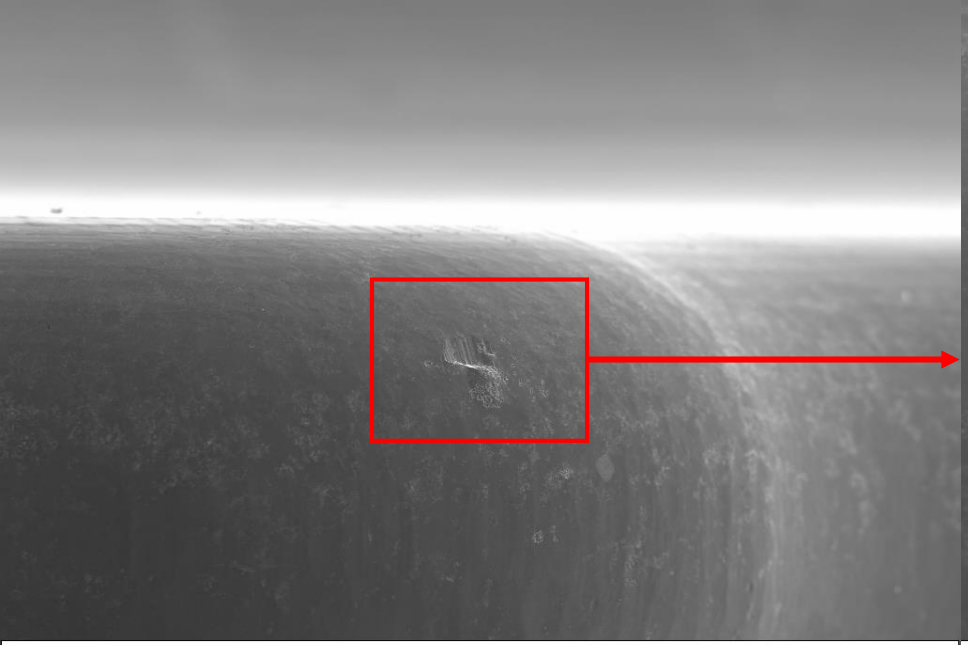
Machining marks

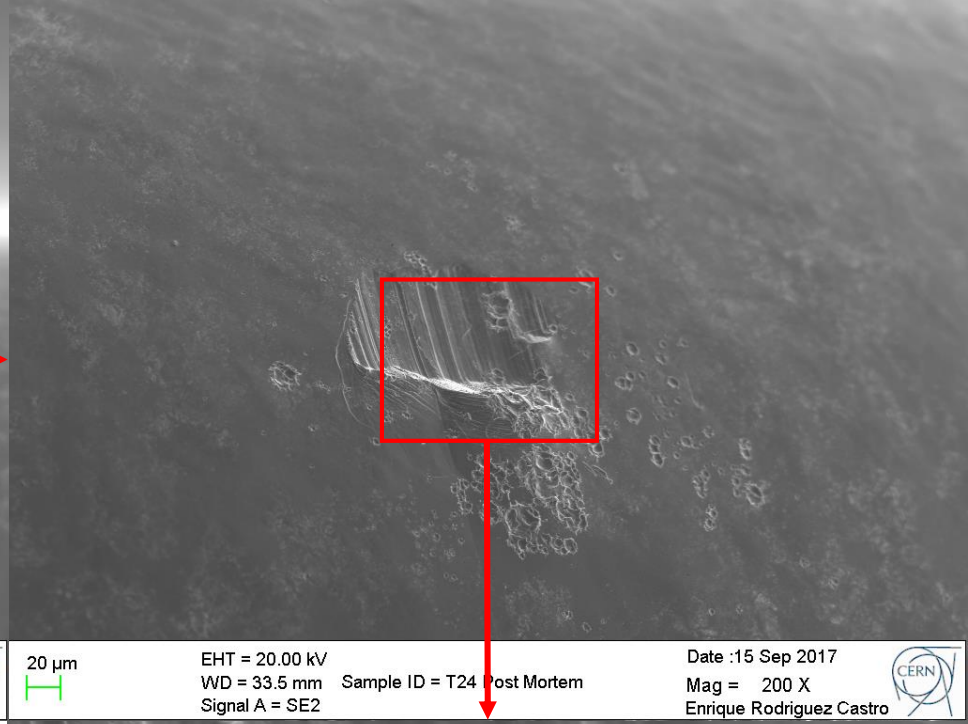


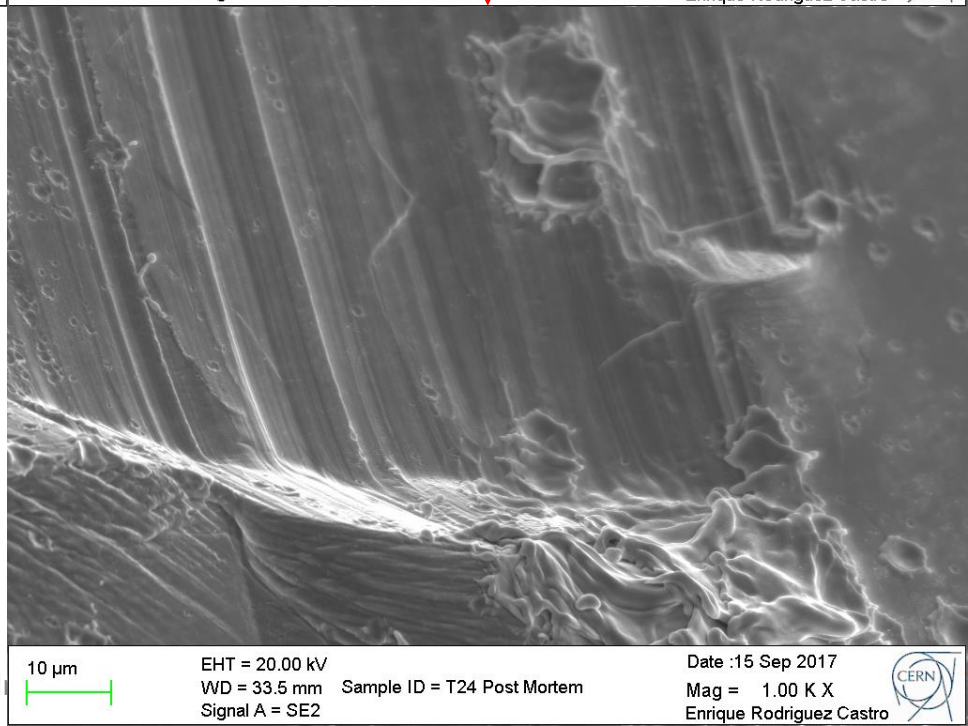

100 µm
EHT = 20.00 kV
WD = 33.6 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :15 Sep 2017
Mag = 50 X
Enrique Rodriguez Castro
CERN


20 µm
EHT = 20.00 kV
WD = 33.6 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :15 Sep 2017
Mag = 200 X
Enrique Rodriguez Castro
CERN



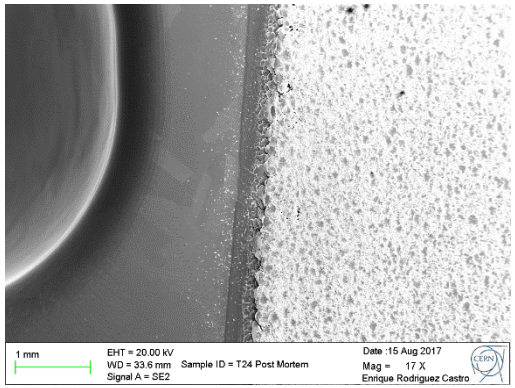

100 µm
EHT = 20.00 kV
WD = 33.5 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :15 Sep 2017
Mag = 50 X
Enrique Rodriguez Castro

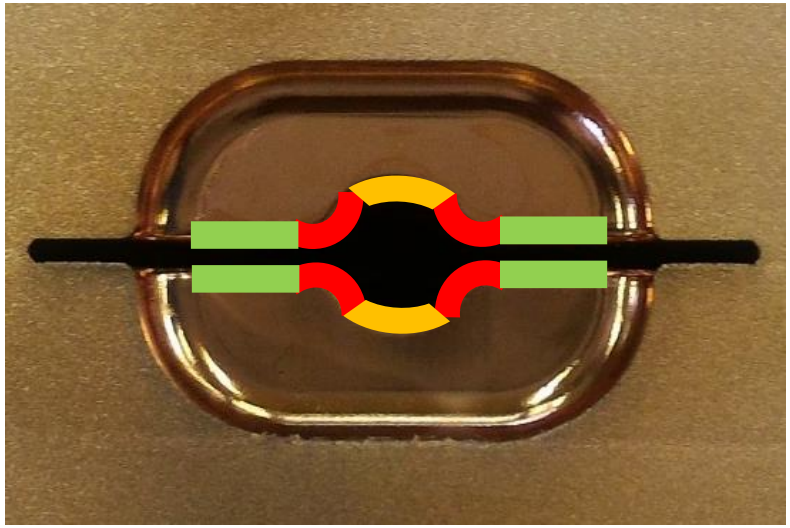

20 µm
EHT = 20.00 kV
WD = 33.5 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :15 Sep 2017
Mag = 200 X
Enrique Rodriguez Castro


10 µm
EHT = 20.00 kV
WD = 33.5 mm
Signal A = SE2
Sample ID = T24 Post Mortem
Date :15 Sep 2017
Mag = 1.00 K X
Enrique Rodriguez Castro

Summary of observation

- Number of BD craters is higher at beginning of the structure
- Defects with high accumulation of BD craters, and others with no BD craters
- Brazing:
  - filler material has flown into the structure.
  - BD craters close to the brazing.
  - splashes of filler material all around the joint region
- Iris:
  - BD accumulation on nose than centre or straight part of the iris





Conclusions

- New equipment and new tools are being added
- Importance of cleanness on the process:
  - Machining
  - Assembly
  - Testing
- Post mortem on RF and DC important tool on BD understanding

Thank you for you attention

Questions?

Recent microscopy at CERN

Enrique Rodríguez Castro