



# Recent microscopy at CERN

*Enrique Rodríguez Castro*



<b>Microscopy at CERN</b>	<b>Introduction</b>	<b>Equipment</b>	OM	
			SEM	
			FIB-SEM	
<b>Microscopy for CLIC</b>	<b>Production</b>	<b>Machining</b>		
		<b>Pollution</b>		
	<b>RF and DC Studies</b>	<b>Post-mortem</b>	CLIC-AS	Crab cavity
				T24 Open
			LES	Materials tests
			Cameras	
			Size vs Gradient	



**Microscopy at CERN**

**Introduction**

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SEM

FIB-SEM

**Microscopy for CLIC**

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**RF and DC Studies**

**Post-mortem**

CLIC-AS

Crab cavity

T24 Open

LES

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Cameras

Size vs Gradient



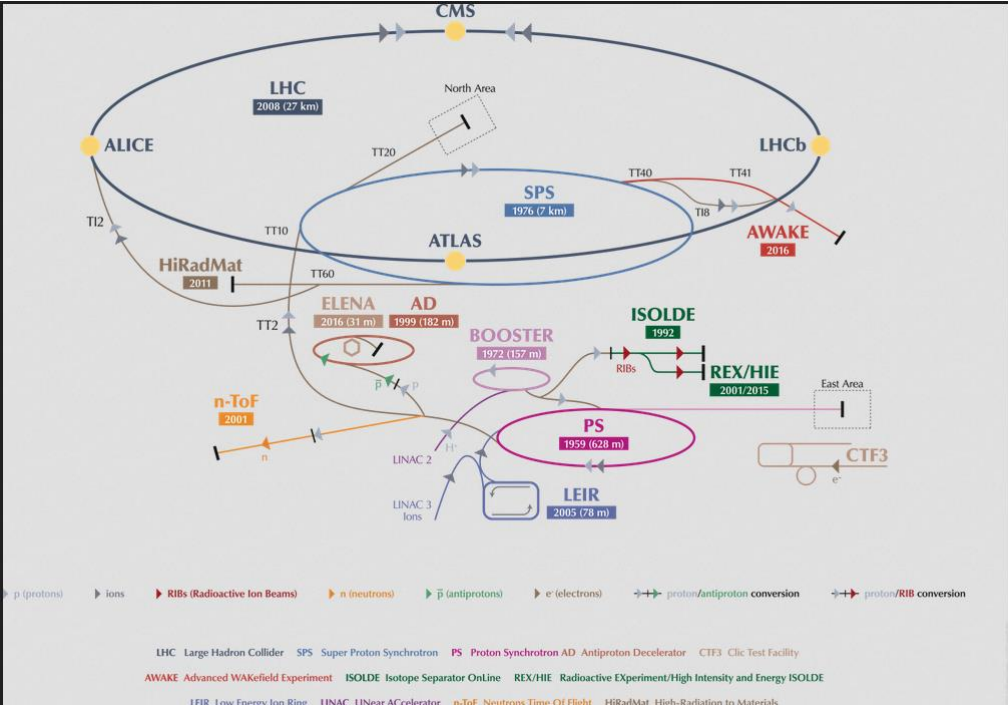
# Microscopy at CERN



ENGINEERING DEPARTMENT



MME Mechanical & Materials Engineering



## 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	7.8x–1,280x
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)	

## 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

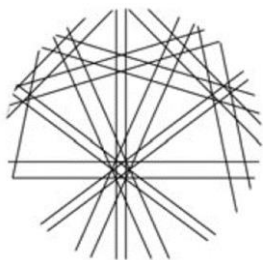
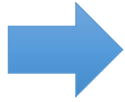
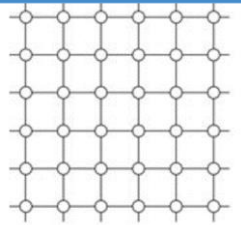


SEM Performance	
Column	Crossbeam 540 (Gemini® II column)
Resolution	High resolution configuration: 0.7 nm at 30 kV
Acceleration Voltage	0.02 - 30 kV
Magnification	12x - 2,000,000x
FIB Performance	
Source	Gallium Ion
Resolution	3nm at 30kV
Acceleration Voltage	0.5 – 30 kV
Magnification	300x - 500,000x

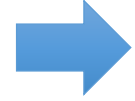


# Mis-orientation and dislocation

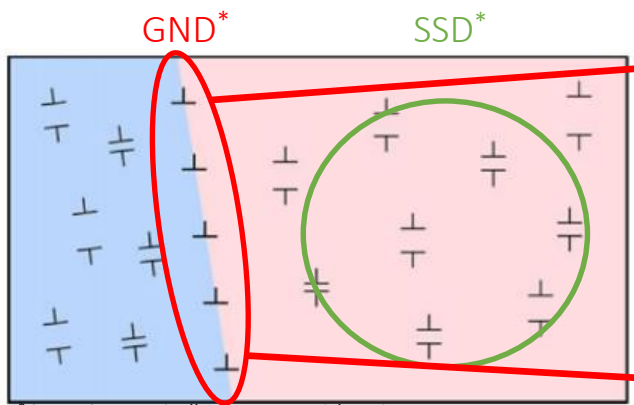
Perfect crystal



Theoretical diffraction pattern



KAM = 0



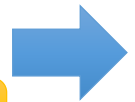
GND\*

SSD\*



Local misorientation

→ Rotation of the pattern



KAM > 0

LAGB : Low Angle Grain Boundaries

KAM : Kernel Average Misorientation

Due to GND

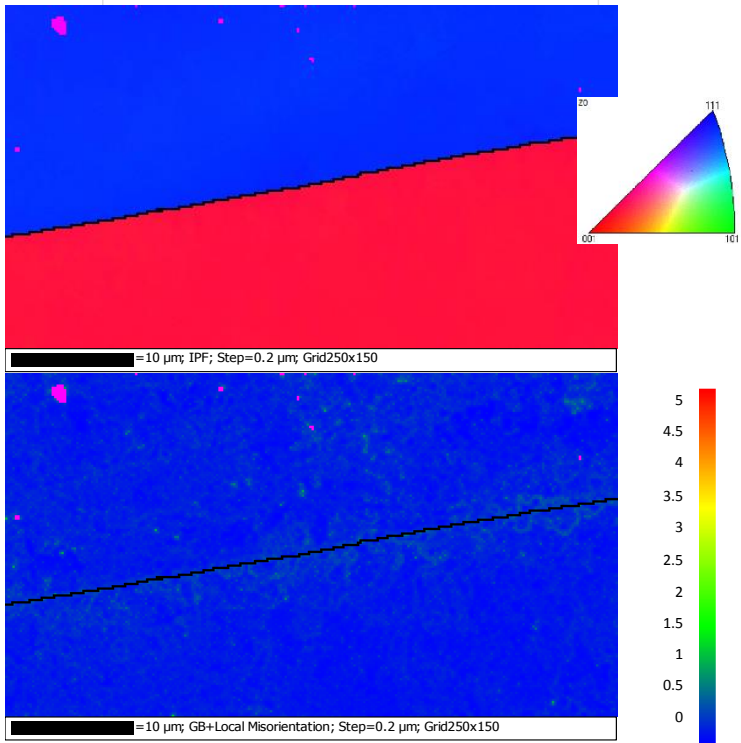
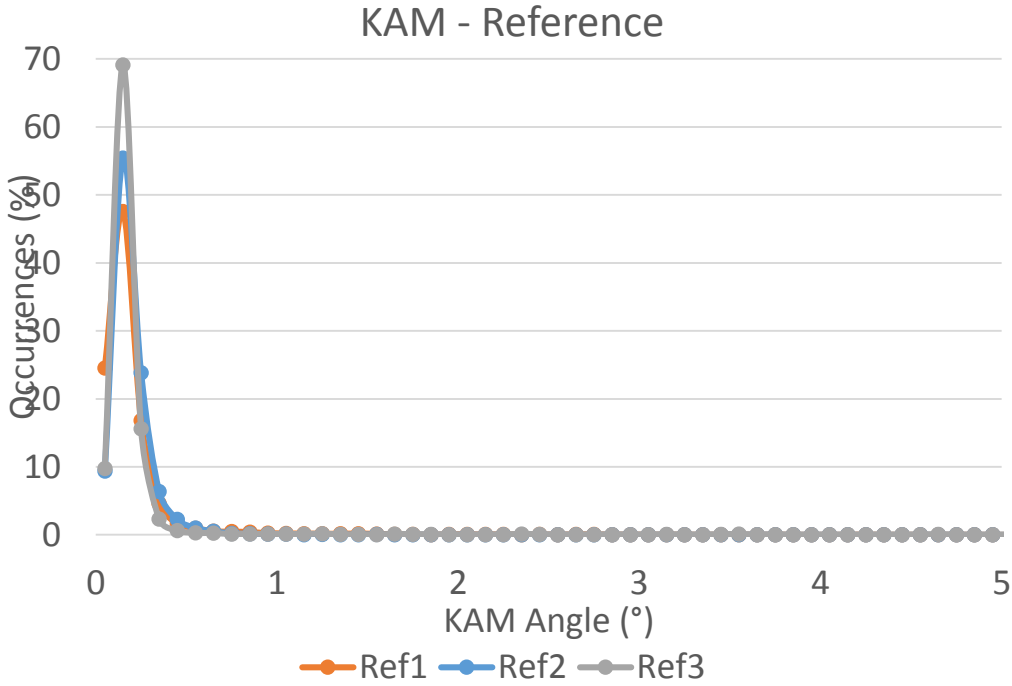
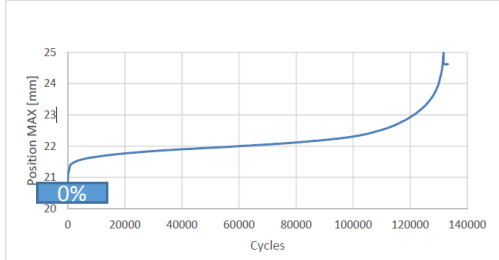
\*GND : Geometrically Necessary Dislocations  
\*SSD : Statistically Stored Dislocations



# Fatigue - Reference (0% LT)



- Narrow distribution
- Low KAM values



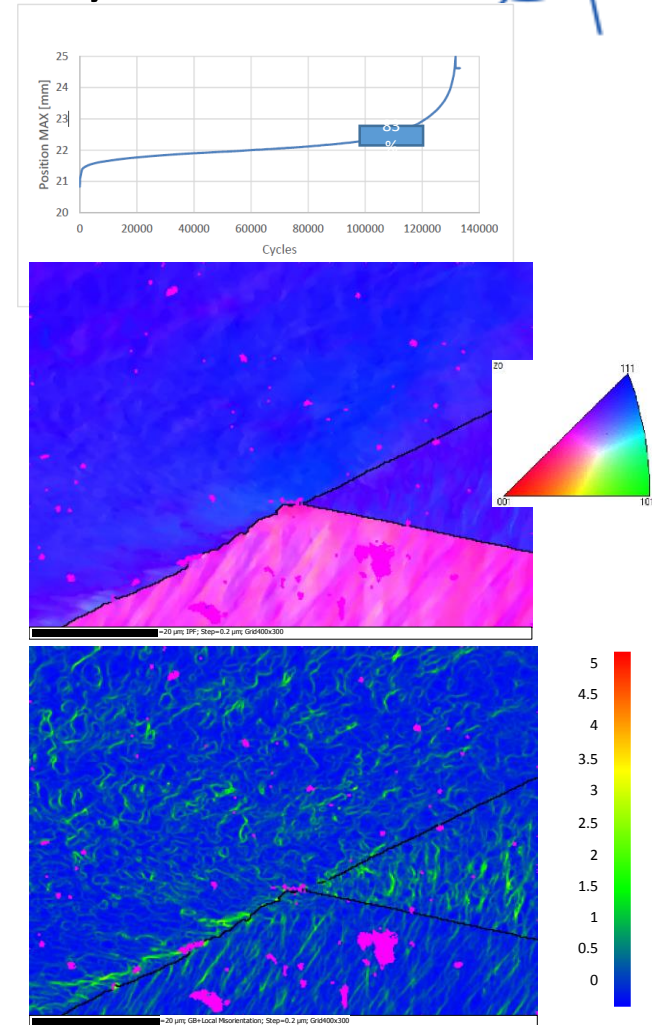
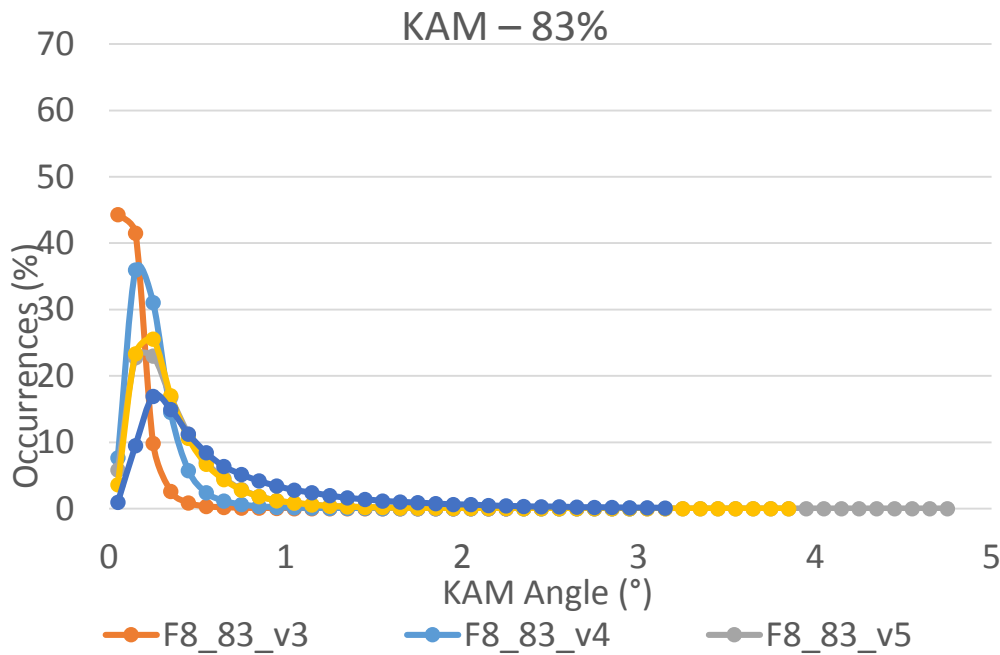




# Fatigued (83% LT)



- Broadening of the distribution
- Shift to higher value
- Higher dispersion

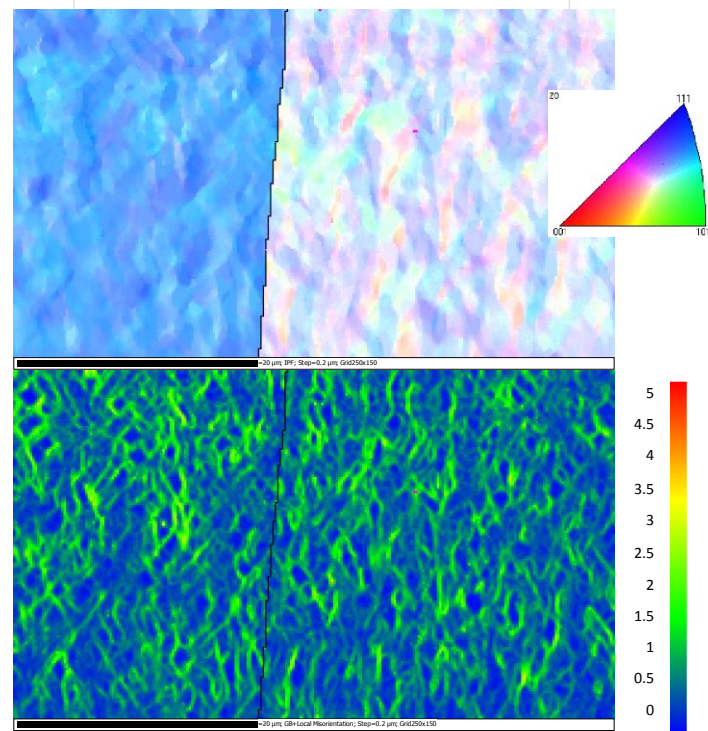
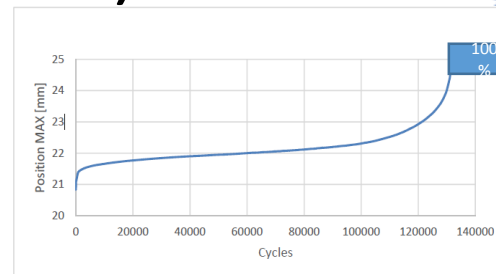
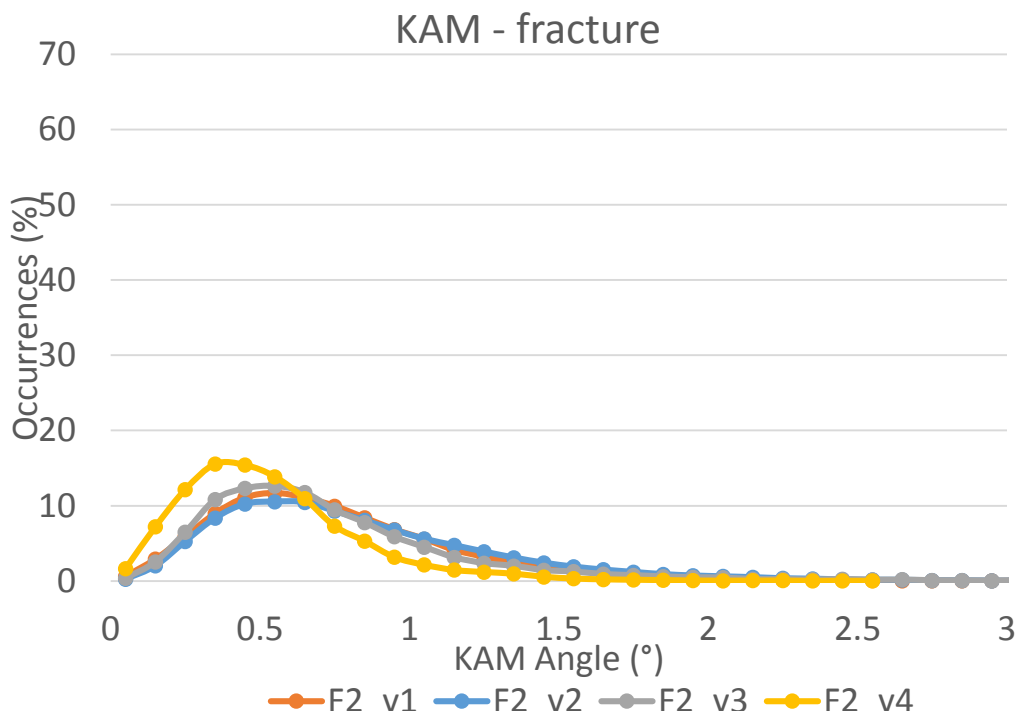


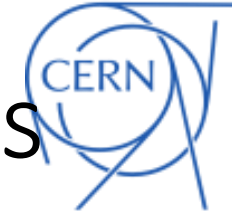


# Fatigued (100% LT)

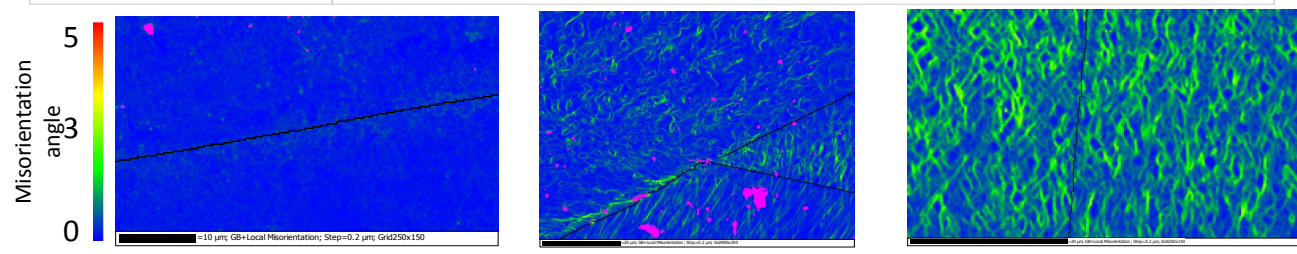
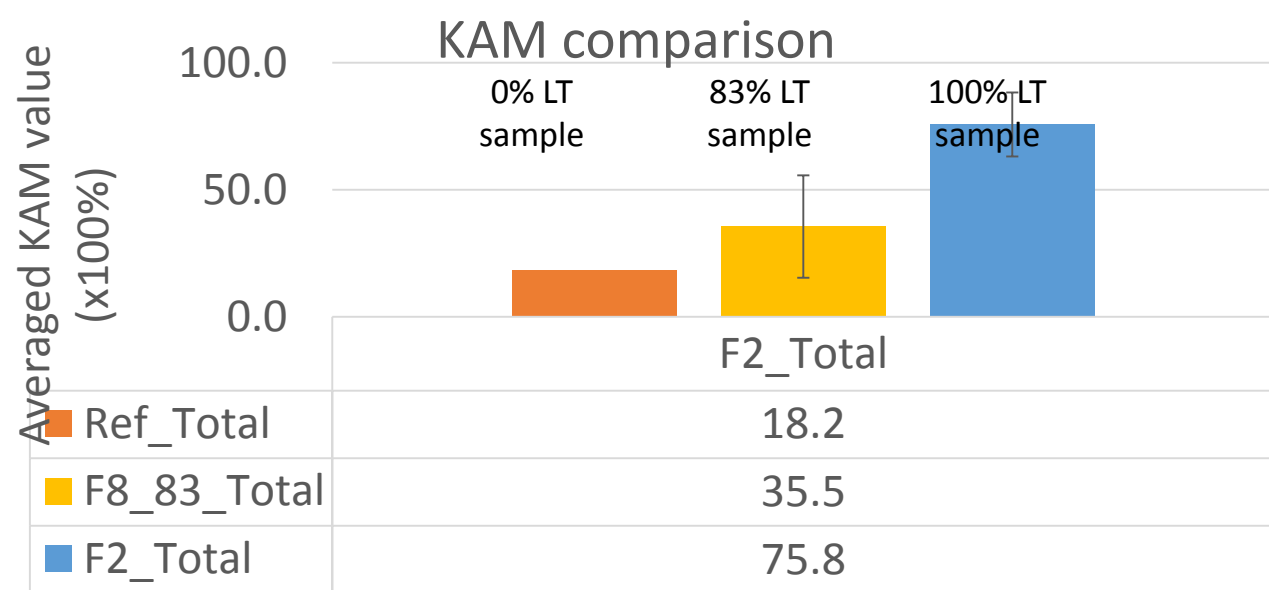


- Strong broadening of the distribution
- Shift to even higher value





# Summary of EBSD observations





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				T24 Open
			LES	Materials tests
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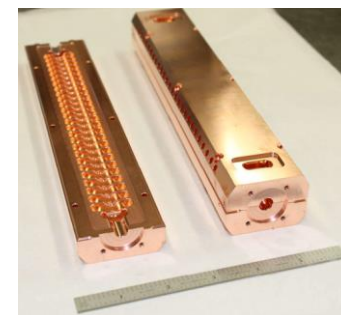
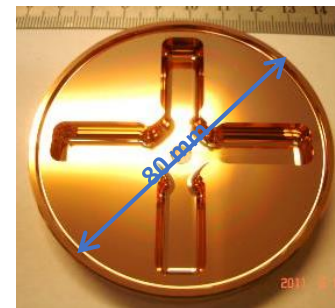
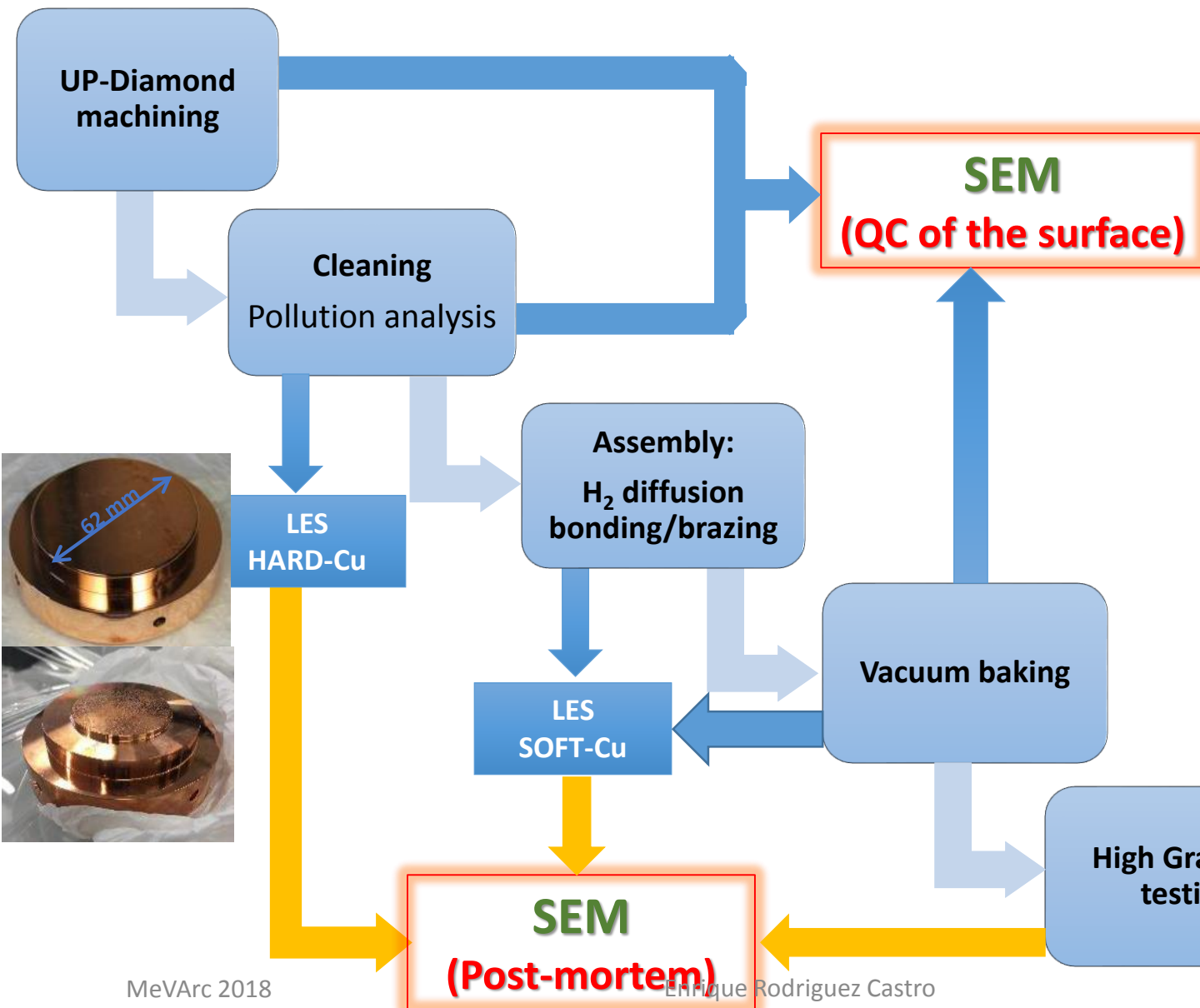
Materials tests

Cameras

Size vs Gradient

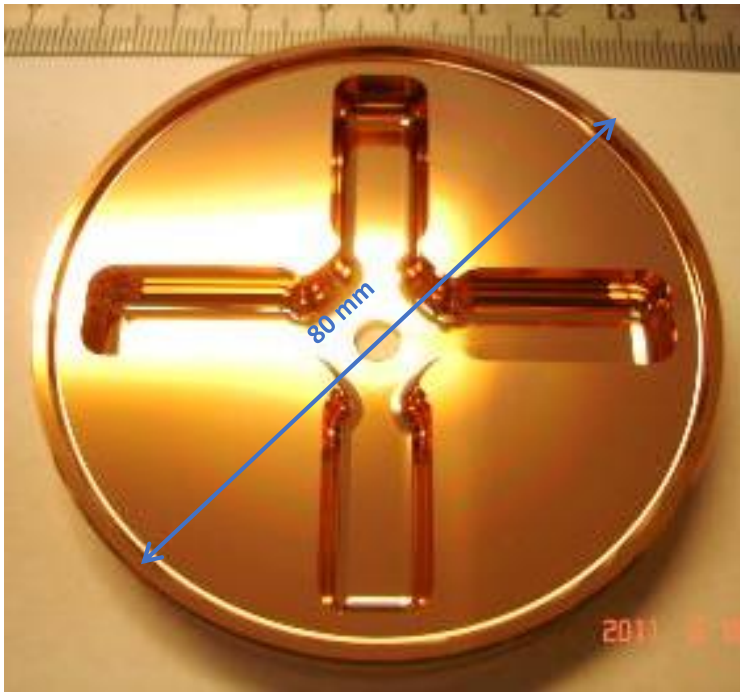
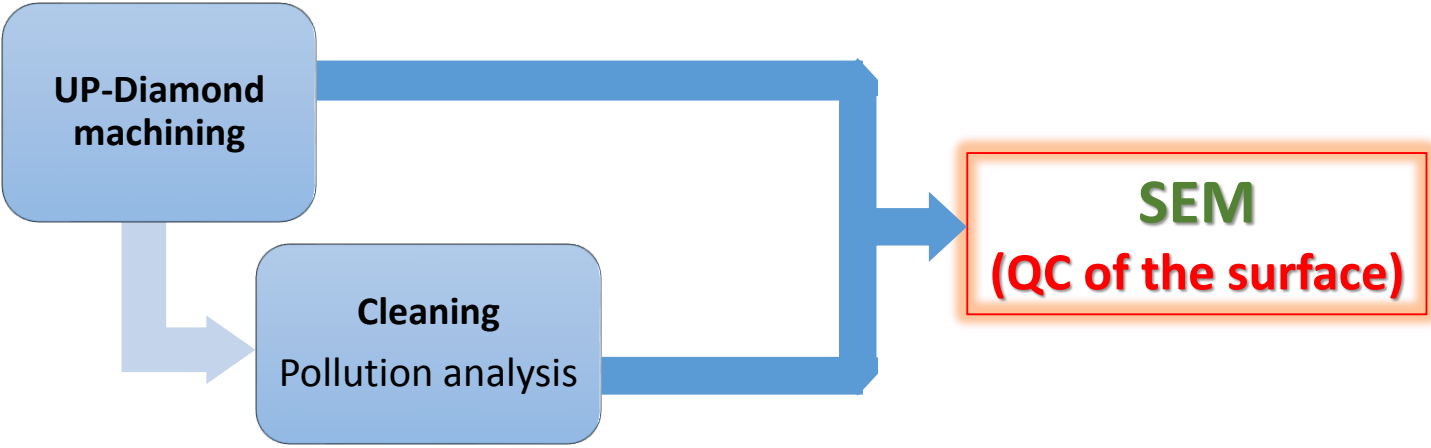


# Microscopy for CLIC





# Microscopy for CLIC

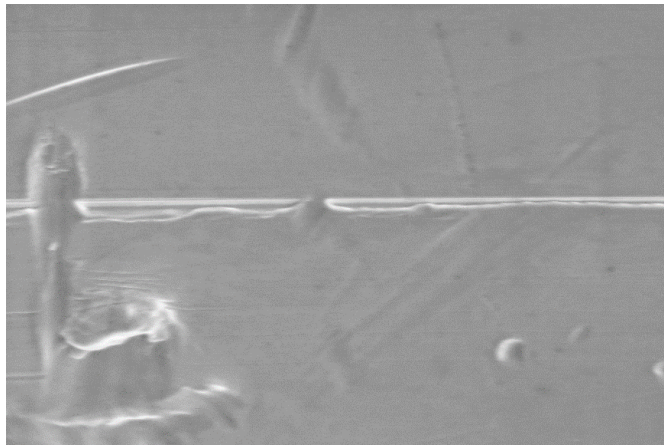




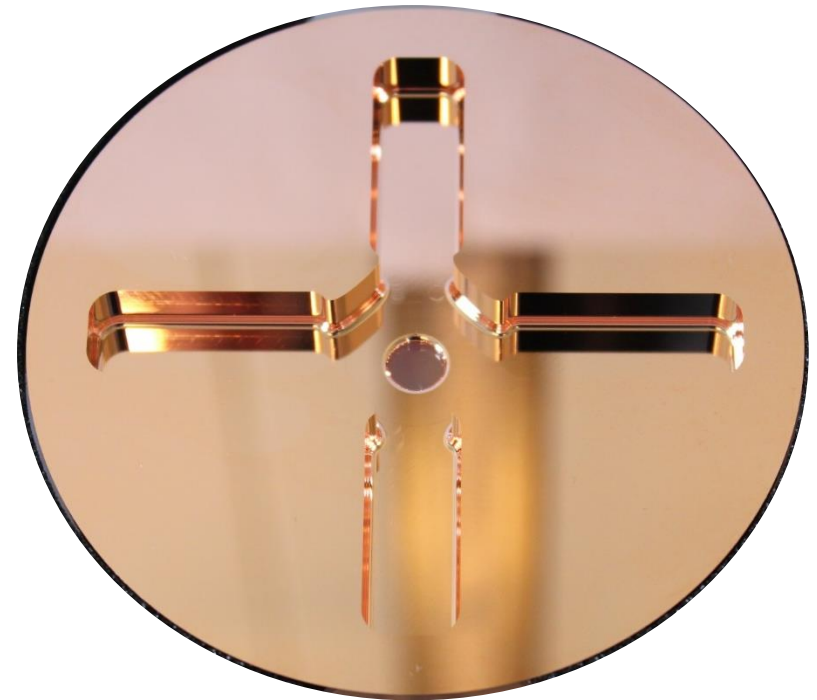
# Machining qualification



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

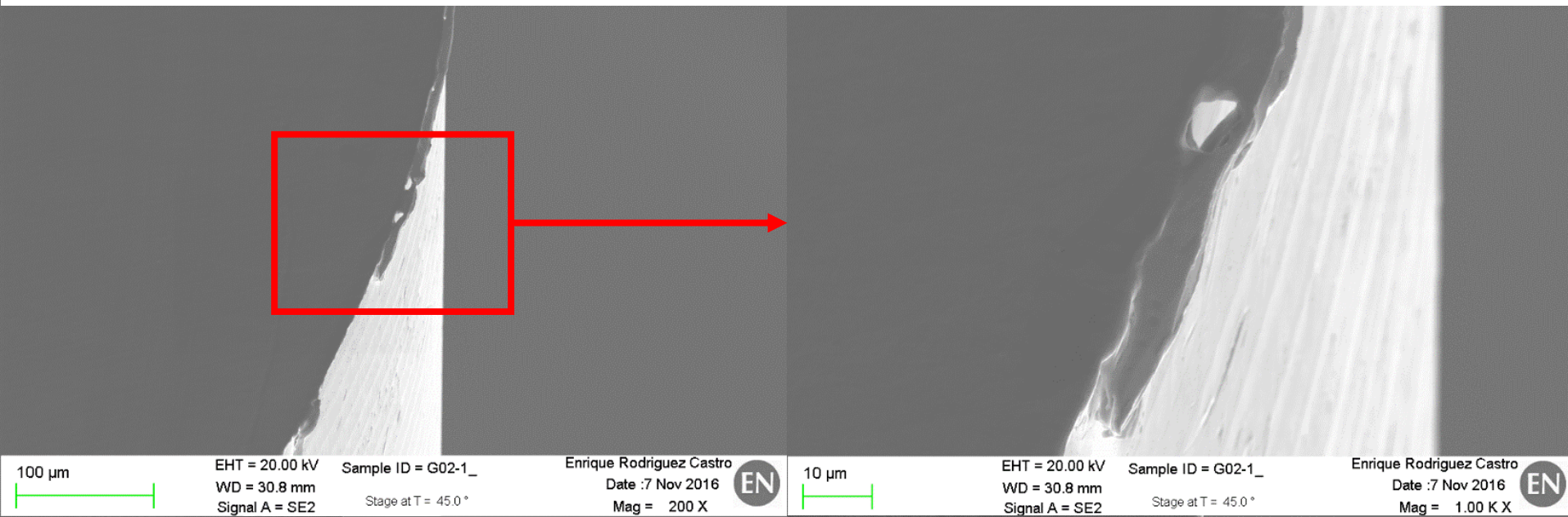


1  $\mu$ m  
EHT = 10.00 kV  
WD = 29.9 mm  
Signal A = SE2  
Sample ID = G02-1\_  
Stage at T = 45.0 °  
Enrique Rodriguez Castro  
Date : 8 Nov 2016  
Mag = 5.00 K X

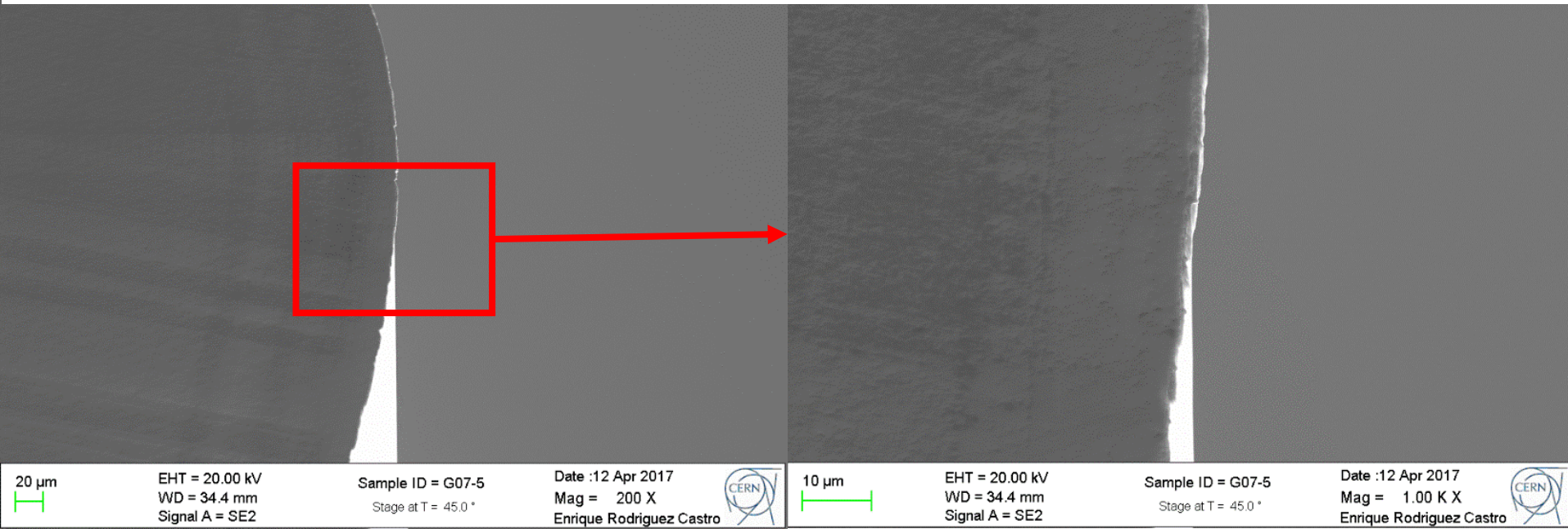




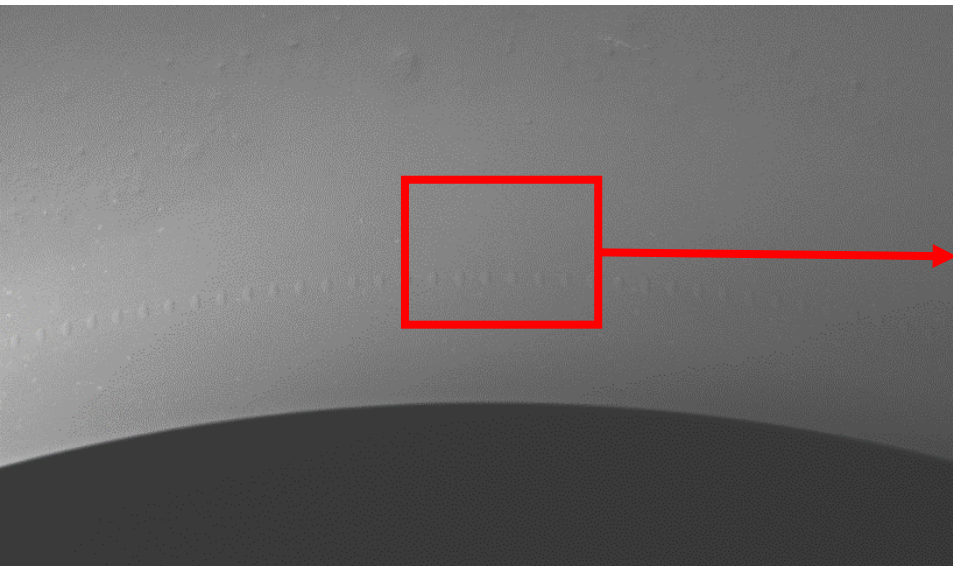
# First observation



# After feedback



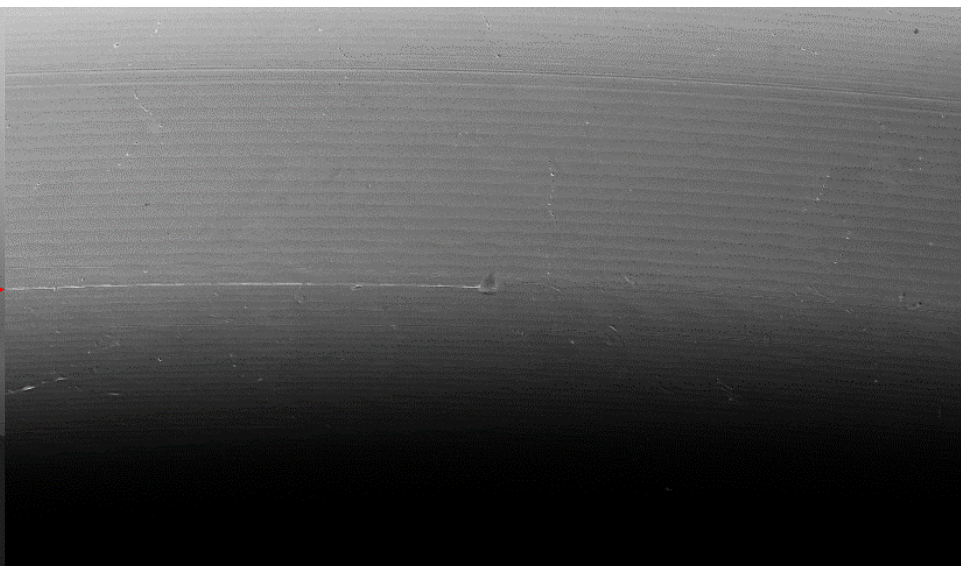
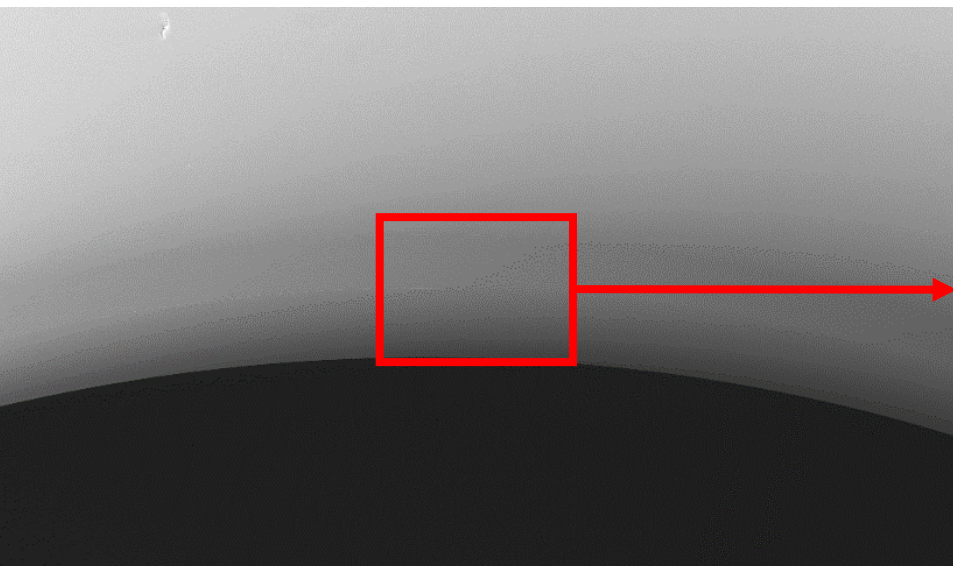
# First observation



100  $\mu\text{m}$   
EHT = 10.00 kV  
WD = 29.3 mm  
Signal A = SE2  
Sample ID = G02-1\_  
Stage at T = 45.0 °  
Enrique Rodriguez Castro  
Date :8 Nov 2016  
Mag = 50 X

100  $\mu\text{m}$   
EHT = 10.00 kV  
WD = 29.9 mm  
Signal A = SE2  
Sample ID = G02-1\_  
Stage at T = 45.0 °  
Enrique Rodriguez Castro  
Date :8 Nov 2016  
Mag = 200 X

# After feedback

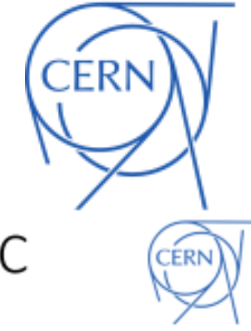


100  $\mu\text{m}$   
EHT = 20.00 kV  
WD = 35.5 mm  
Signal A = SE2  
Sample ID = G07-5  
Stage at T = 45.0 °  
Enrique Rodriguez Castro  
Date :12 Apr 2017  
Mag = 50 X

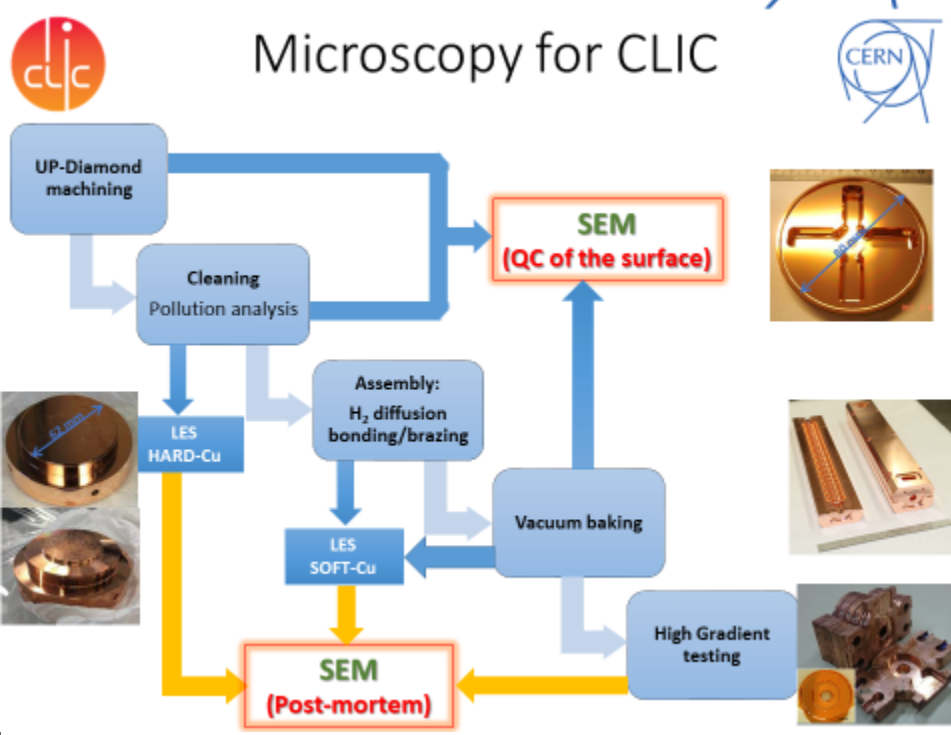
20  $\mu\text{m}$   
EHT = 20.00 kV  
WD = 35.5 mm  
Signal A = SE2  
Sample ID = G07-5  
Stage at T = 45.0 °  
Enrique Rodriguez Castro  
Date :12 Apr 2017  
Mag = 200 X



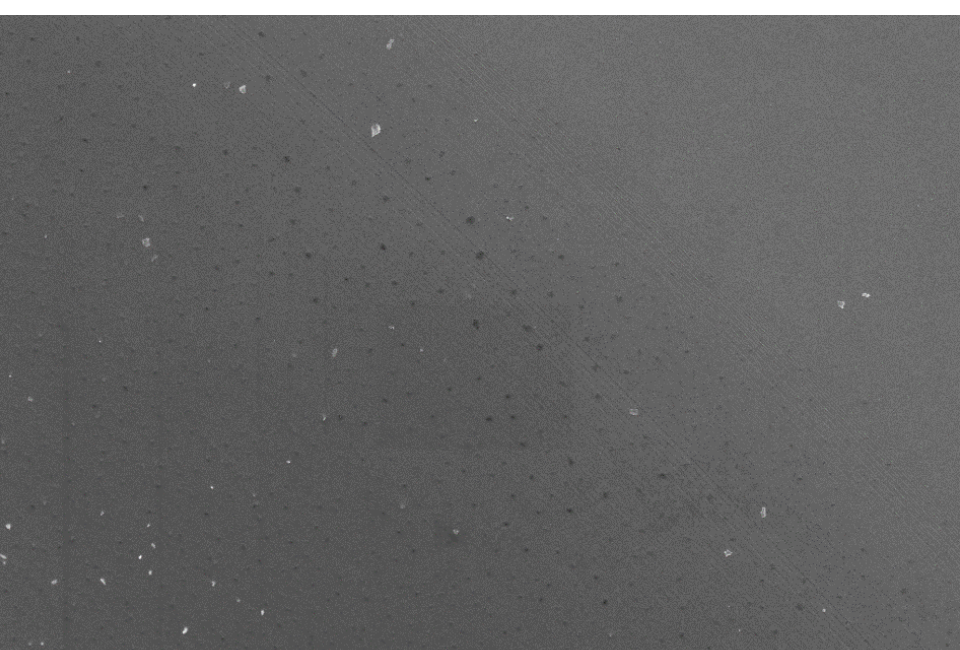
# Pollution Analysis



- 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).



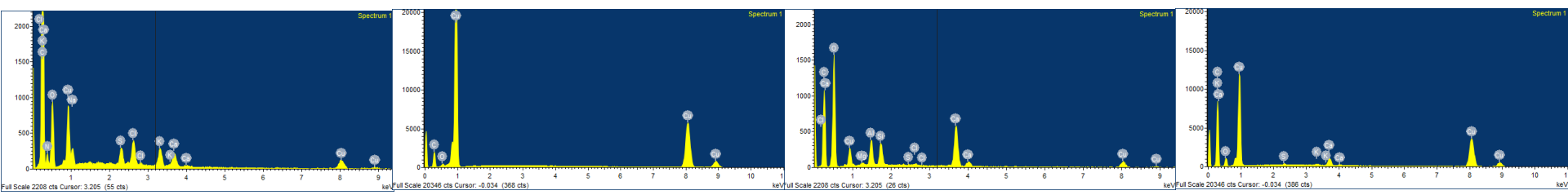
- Witness disc to trace back if any problem is encountered





# APA (Automated Particle Analysis)

- Possibility to create custom classification
  - Ignore non important SOI
  - Enhance those with dangerous contaminants



- Shape recognition.
- From qualitative to quantitative

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



**Microscopy at CERN**

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**Microscopy for CLIC**

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**RF and DC Studies**

**Post-mortem**

CLIC-AS

Crab cavity

T24 Open

LES

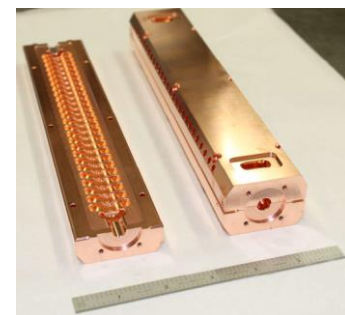
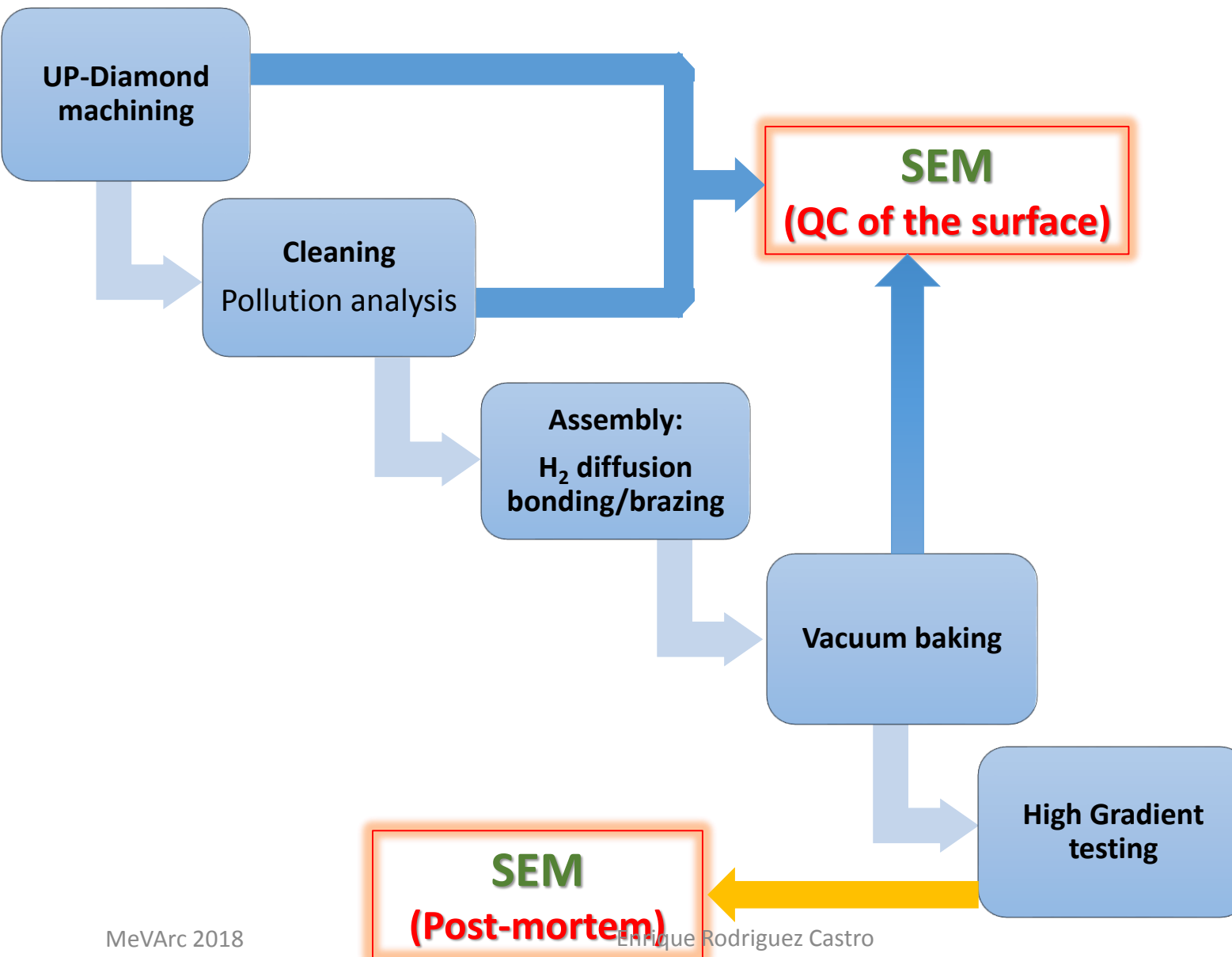
Soft / hard Cu

Cameras

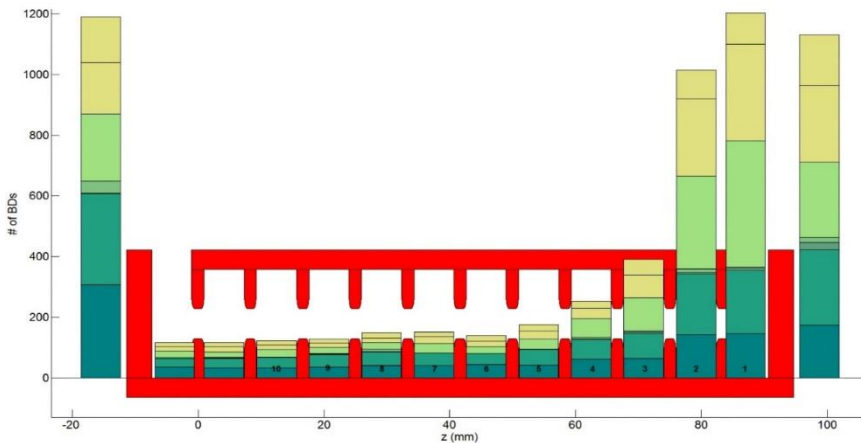
Size vs Gradient



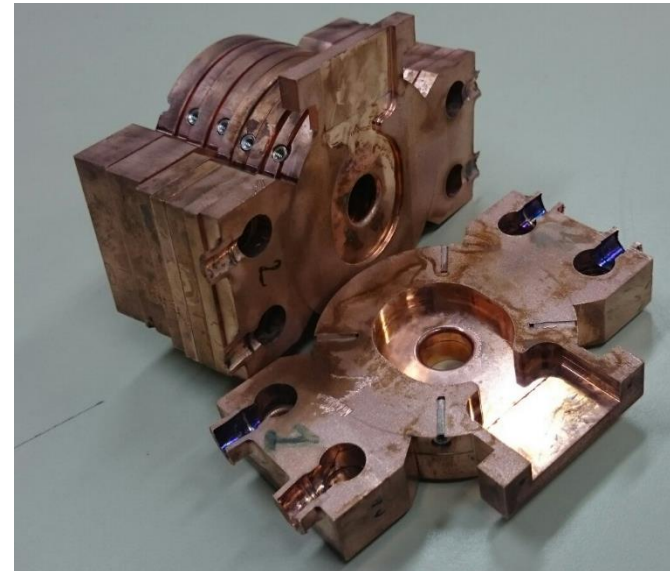
# Microscopy for CLIC



- Not camera system on AS as we do have in DC
- 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.

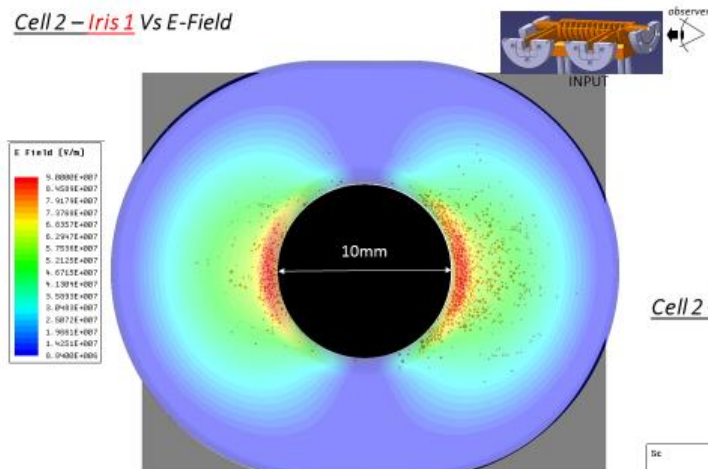




# Crab cavity

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

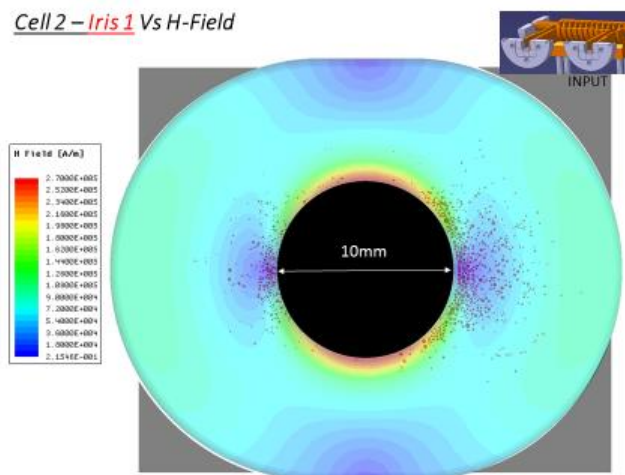
Cell 2 – Iris 1 Vs E-Field



June 2016

Enrique Rodriguez Castro

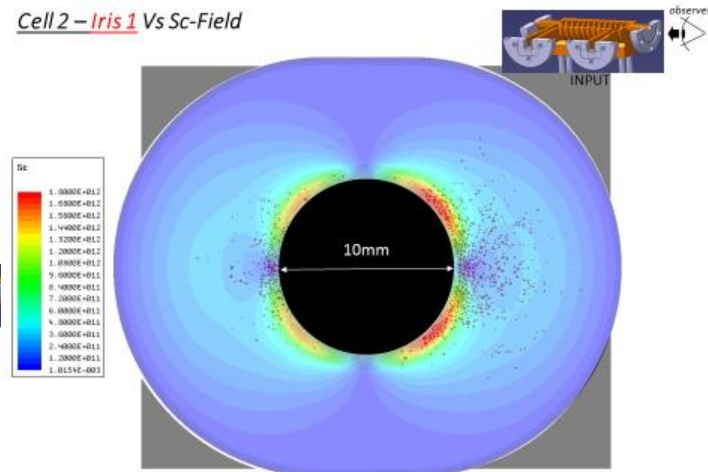
Cell 2 – Iris 1 Vs H-Field



June 2016

Enrique Rodriguez Castro

Cell 2 – Iris 1 Vs Sc-Field



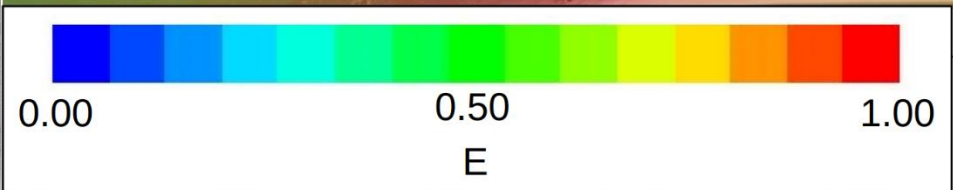
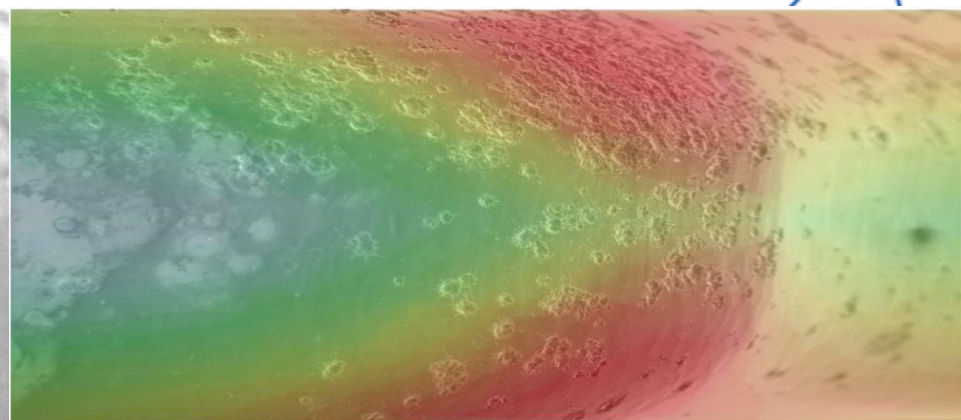
June 2016

Enrique Rodriguez Castro





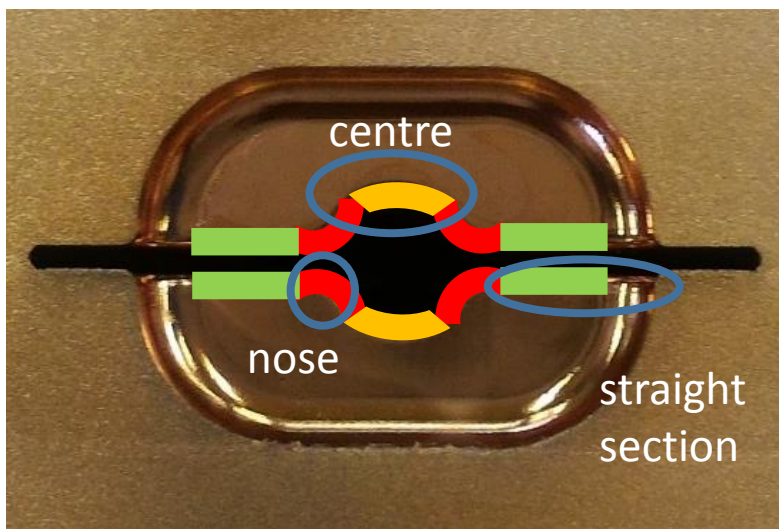
# T24 Open



100  $\mu$ m      EHT = 20.00 kV      Date :15 Sep 2017  
 WD = 34.0 mm      Sample ID = T24 Post Mortem      Mag = 50 X  
 Signal A = SE2      Enrique Rodriguez Castro



- More BD accumulation on **nose** than **center** or **straight part** of the iris, matching the E-field
- BD trend to accumulate in E\_max





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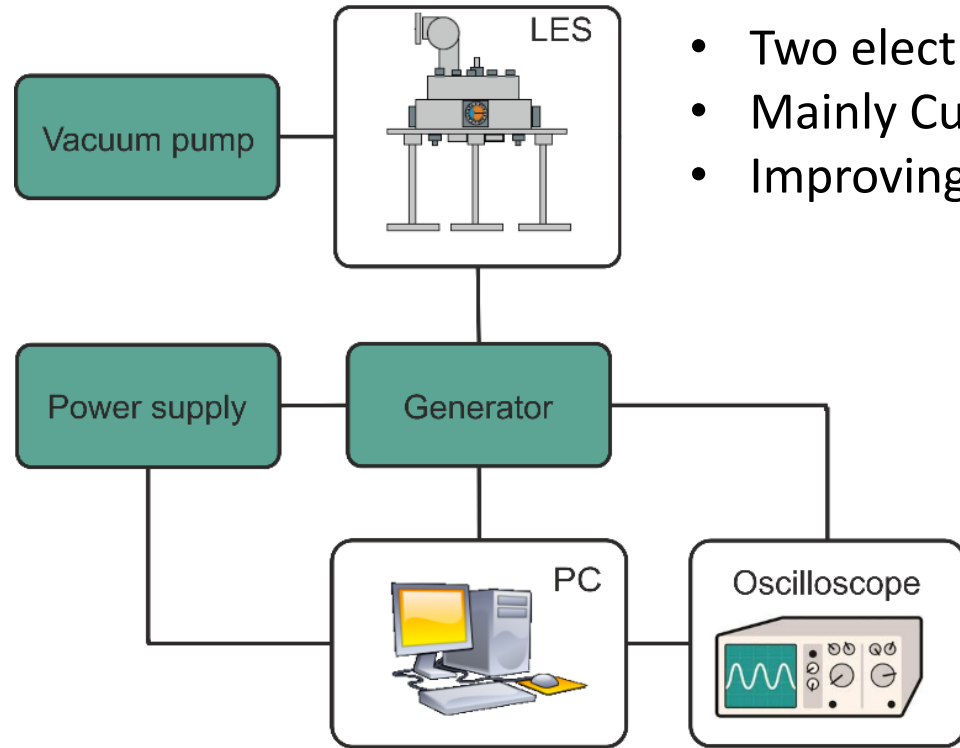
LES

Materials tests

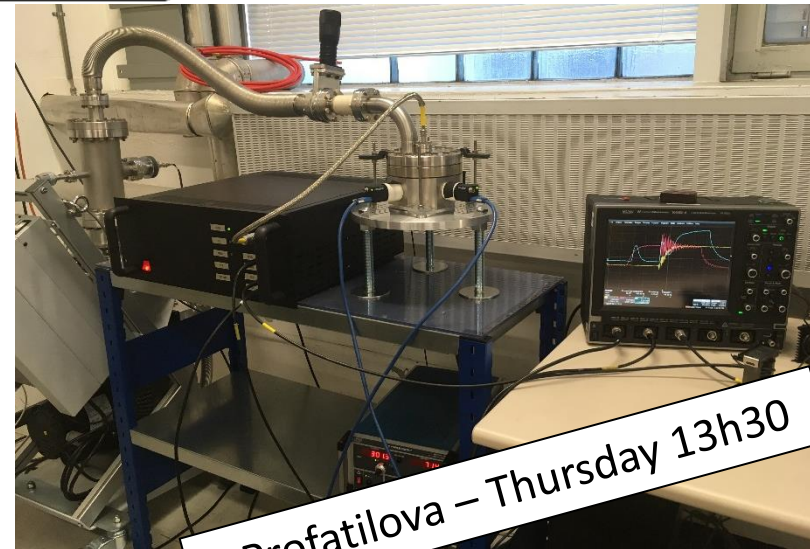
Cameras

Size vs Gradient

- Two electrodes tested under vacuum
- Mainly Cu but also other materials as Nb, SS, CuAg
- Improving the system and the tests



Repetition rate: 2 kHz  
 Pulse length: 500 ns – 1 ms.



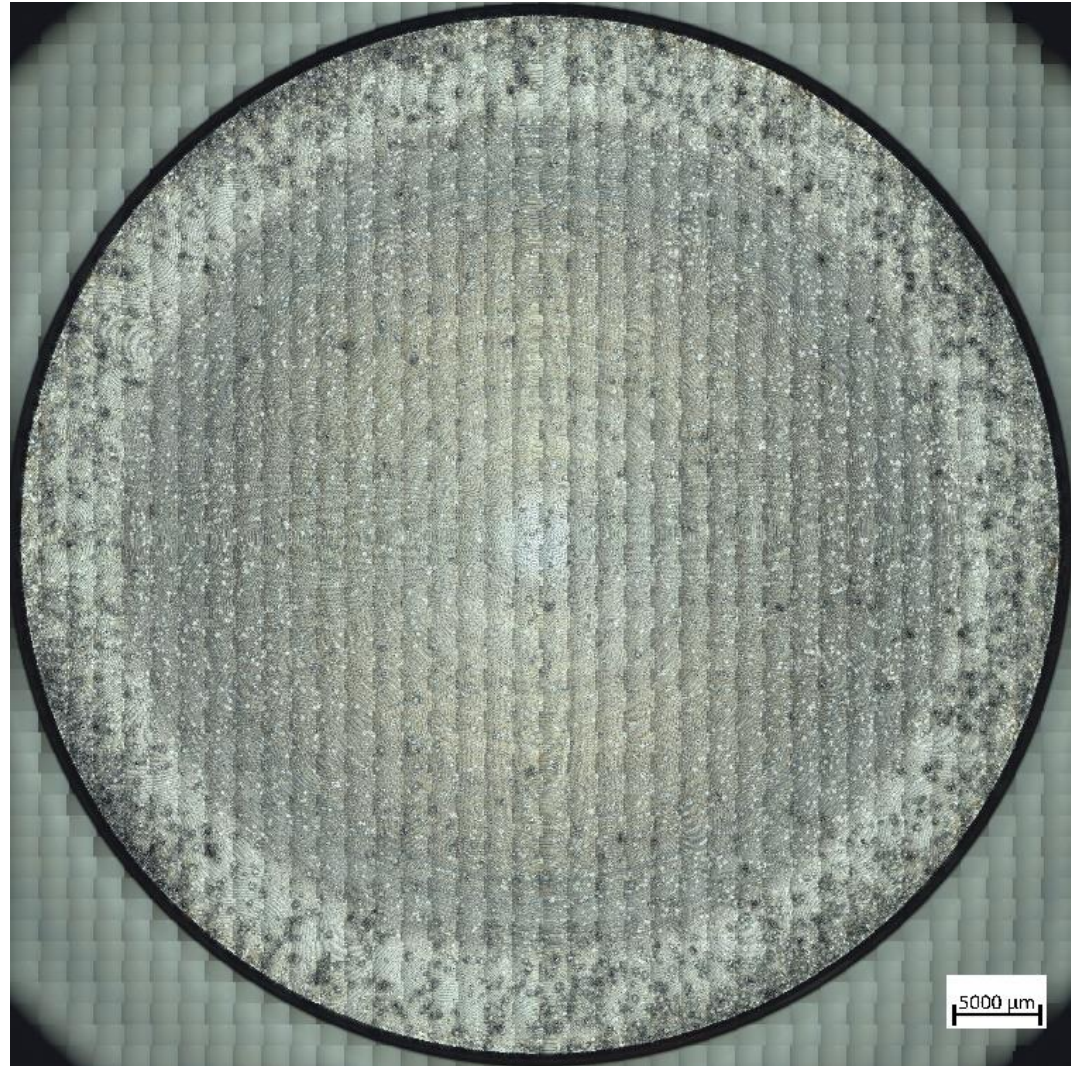
I. Profatilova – Thursday 13h30



# Large Electrode System (LES)



- SS anode tested against 2 different cathode:
  1. CuAg
  2. Fired vacuum Cu

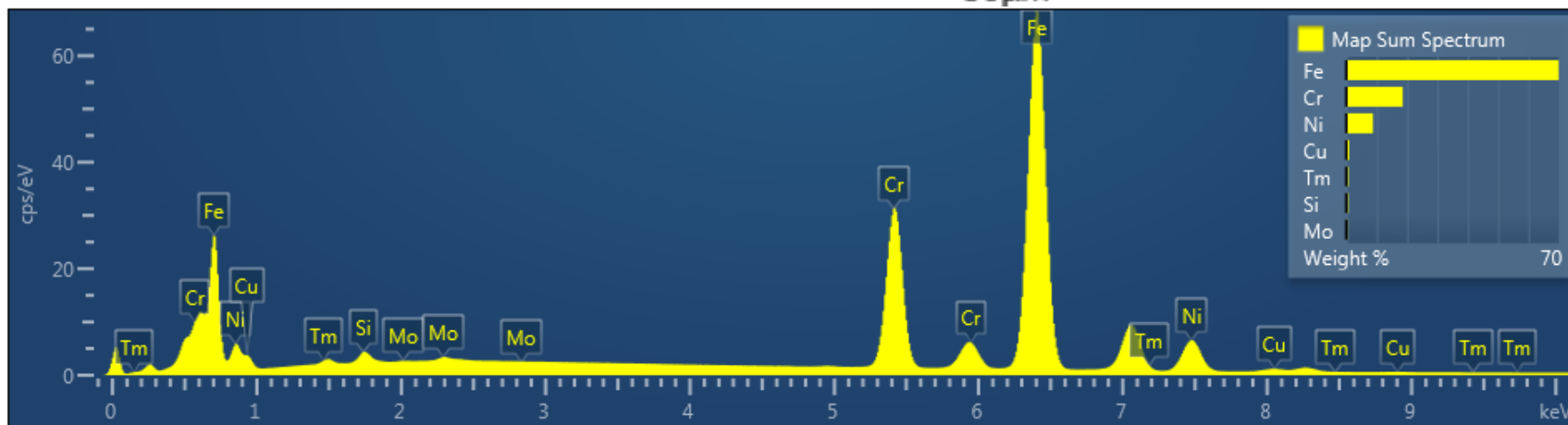
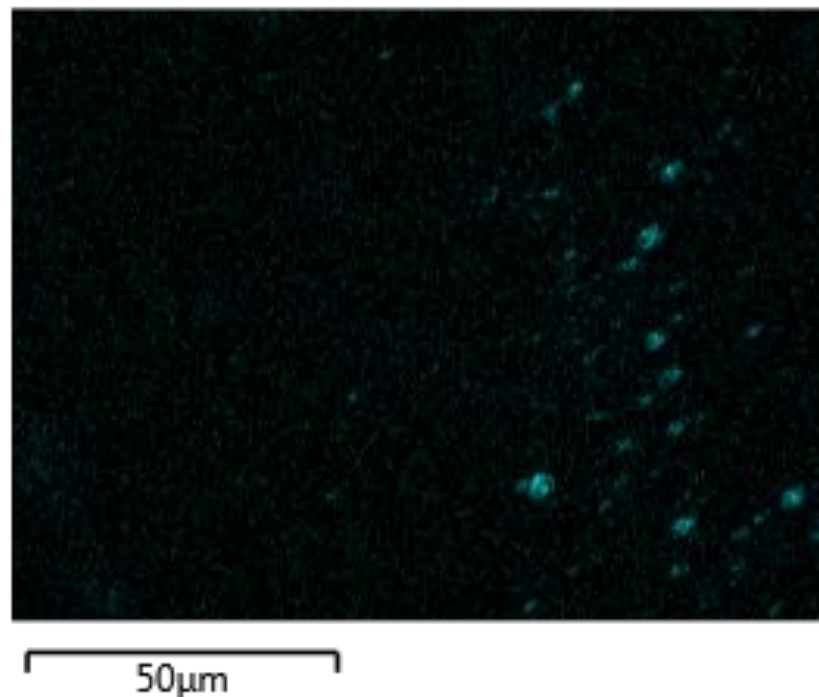
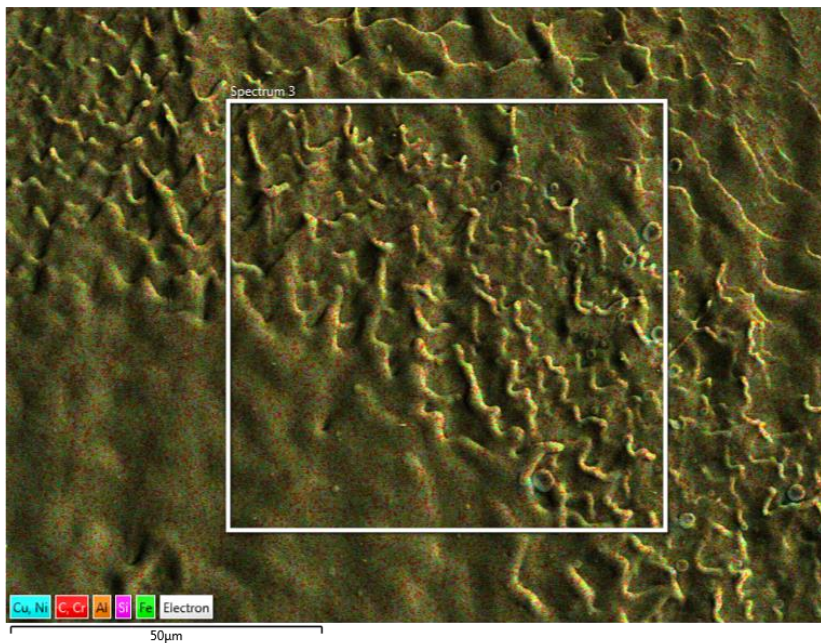




# Large Electrode System (LES)



Cu K series

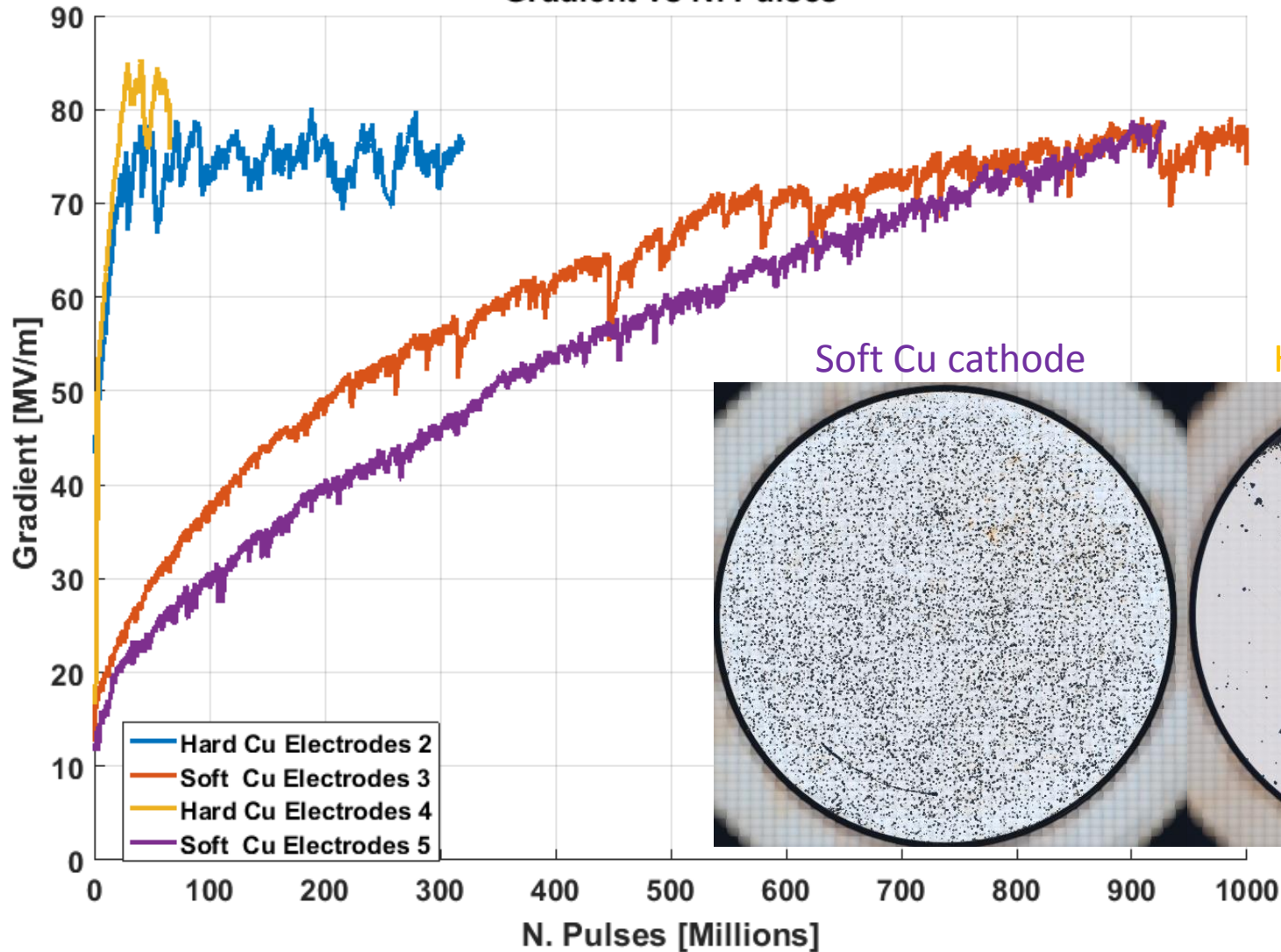




# Large Electrode System (LES)

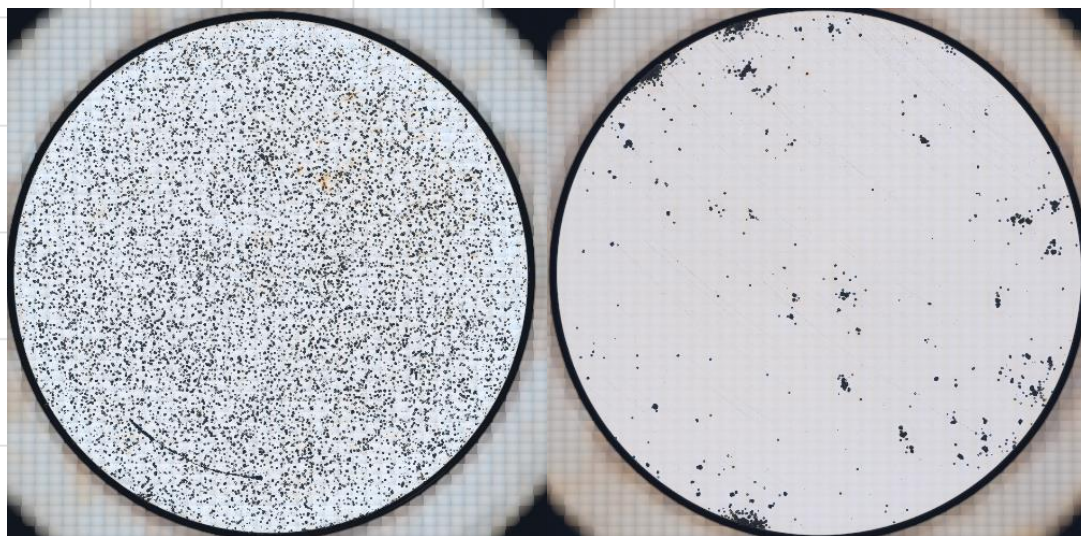


Gradient vs N. Pulses



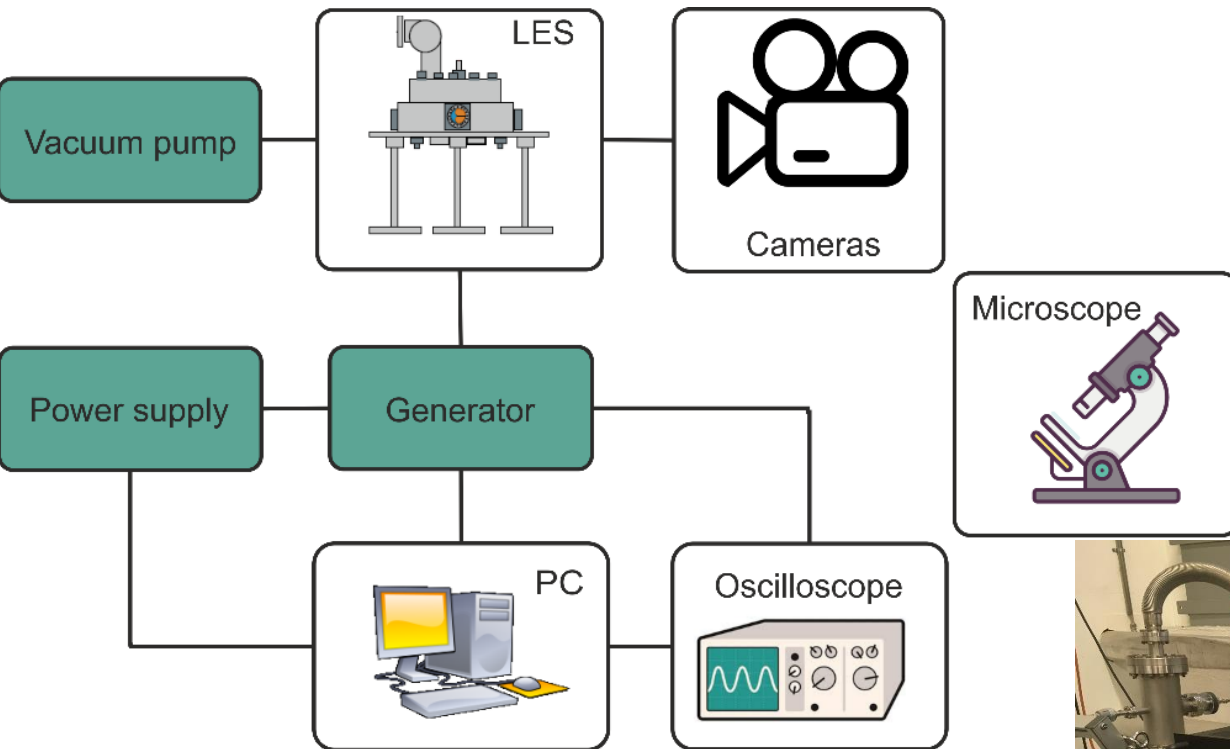
Soft Cu cathode

Hard Cu cathode

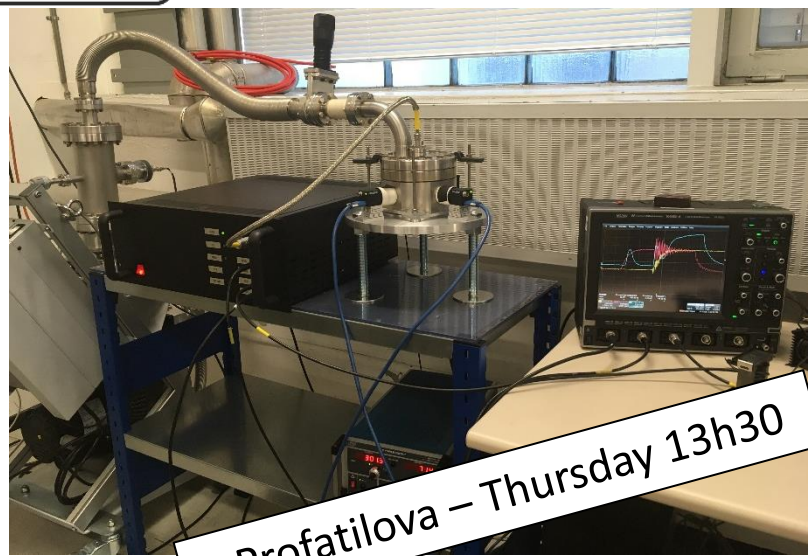




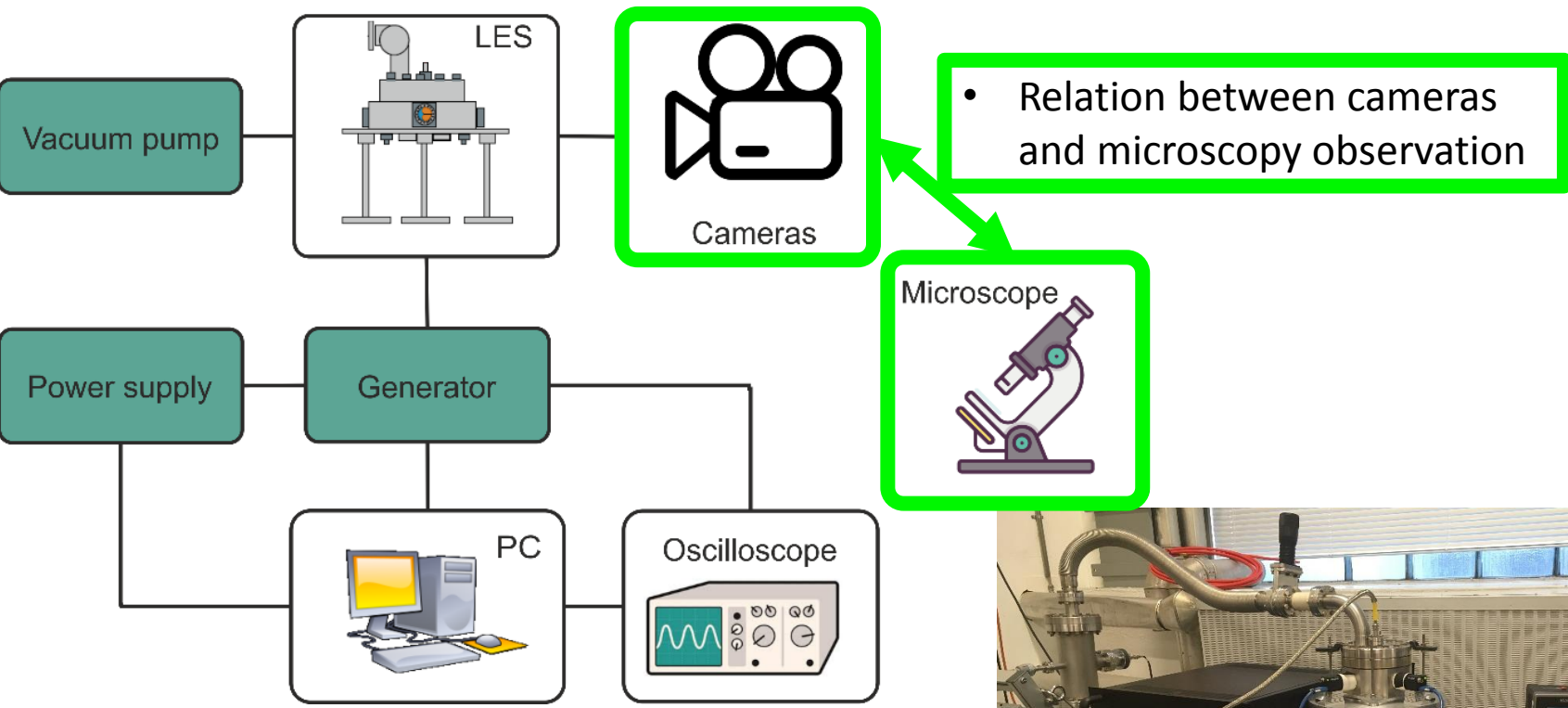
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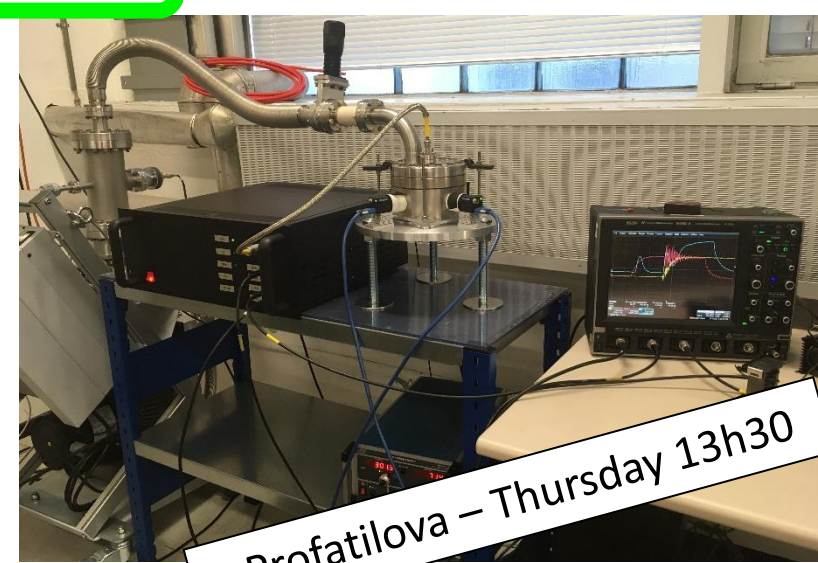
Repetition rate: 2 kHz  
Pulse length: 500 ns – 1 ms.



I. Profatilova – Thursday 13h30



Repetition rate: 2 kHz  
Pulse length: 500 ns – 1 ms.



I. Profatilova – Thursday 13h30





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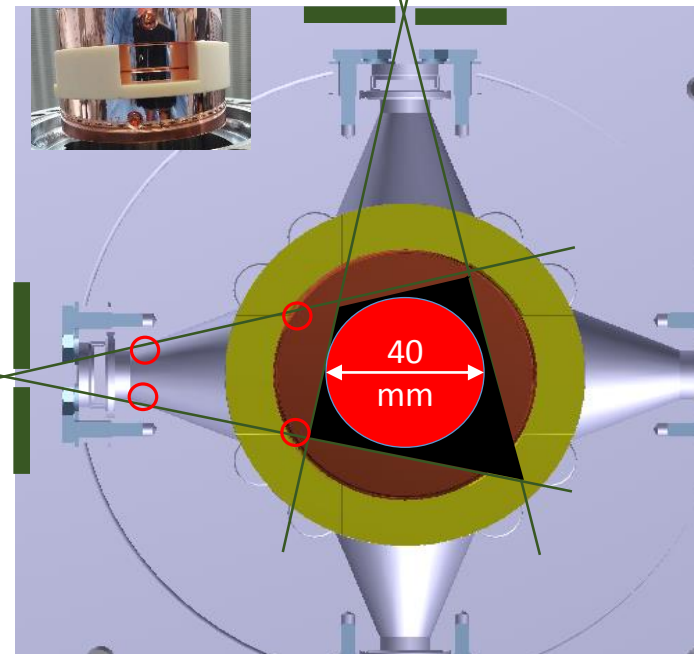
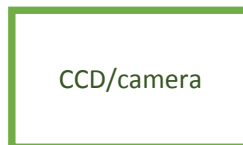
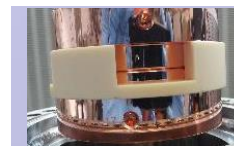


## Motivation:

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



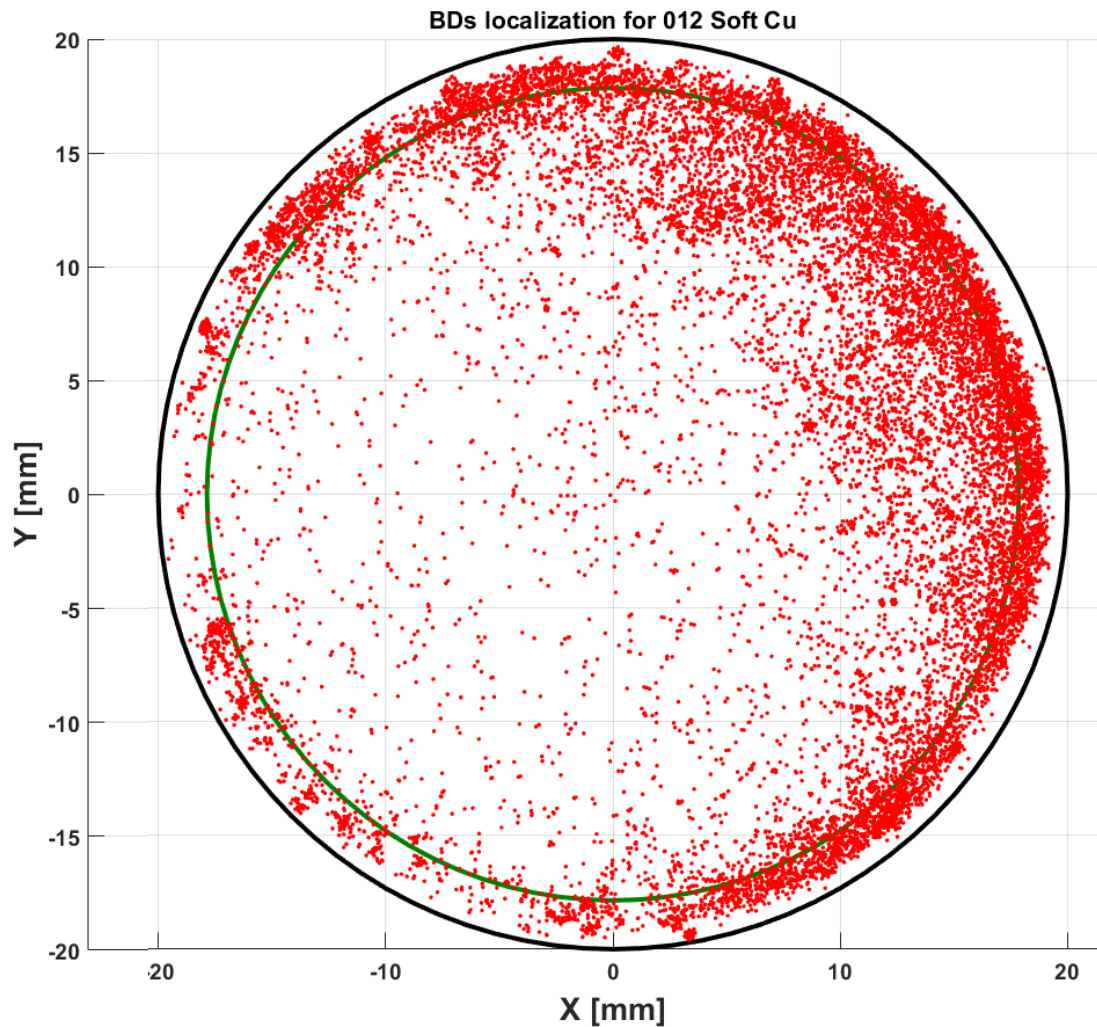
Electrode restrictions



Courtesy  
I. Profatlova and X. Stragier

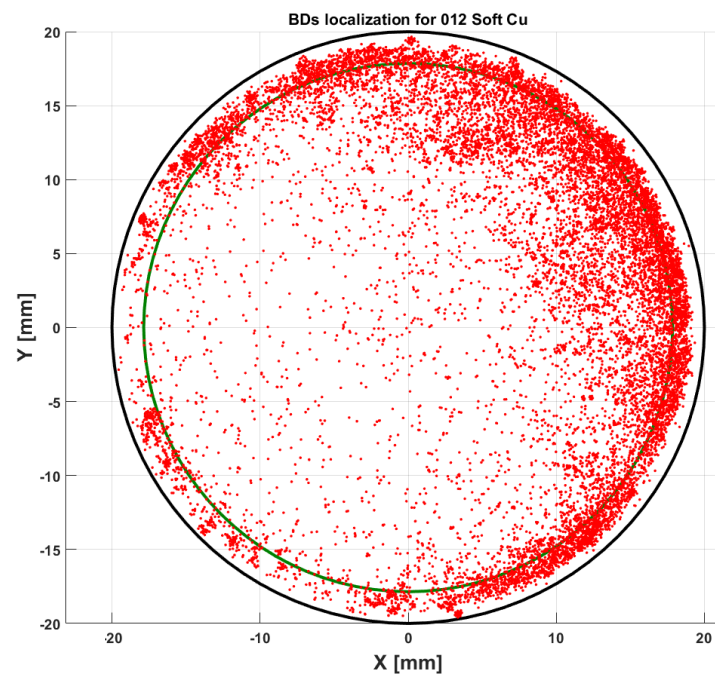
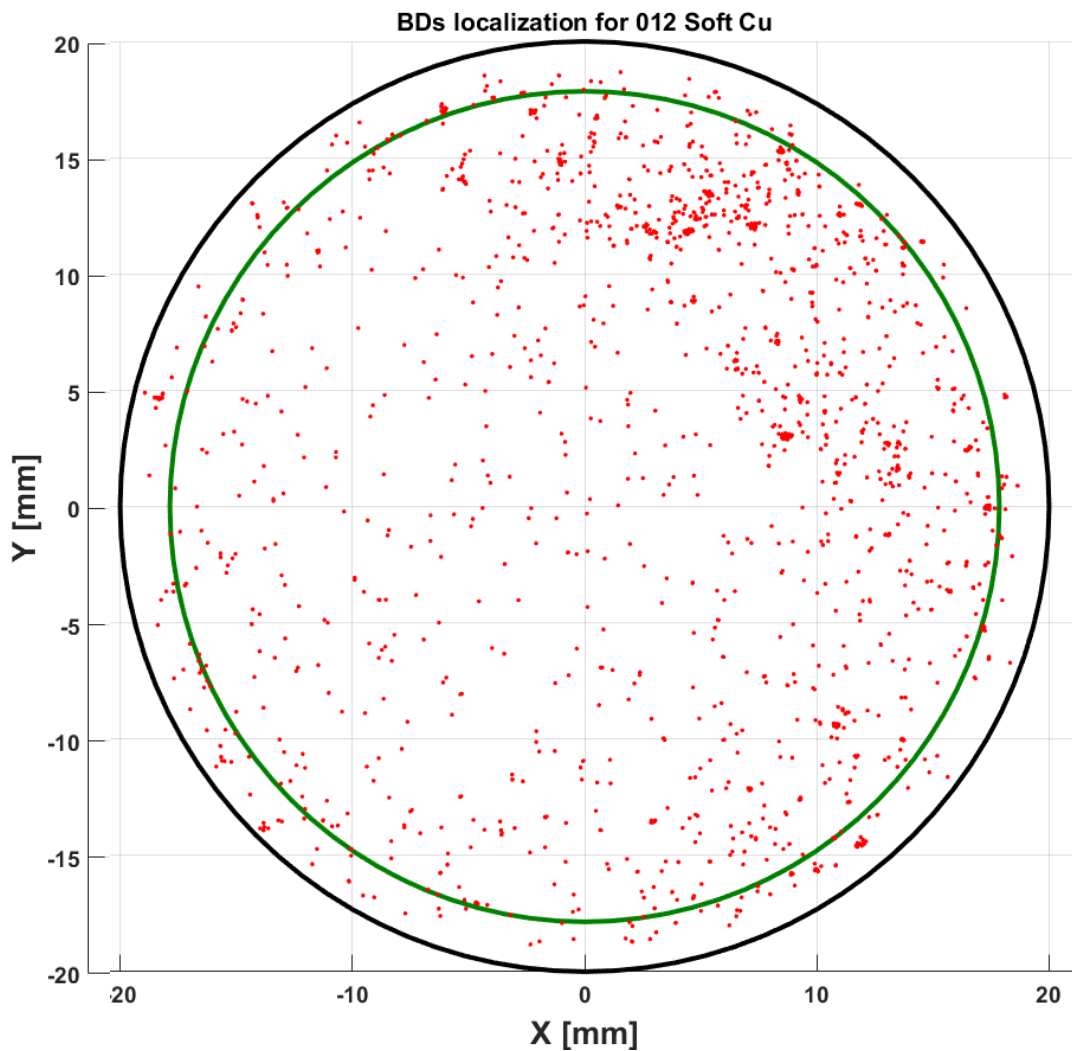


# Large Electrode System (LES)





# Large Electrode System (LES)





# Large Electrode System (LES)

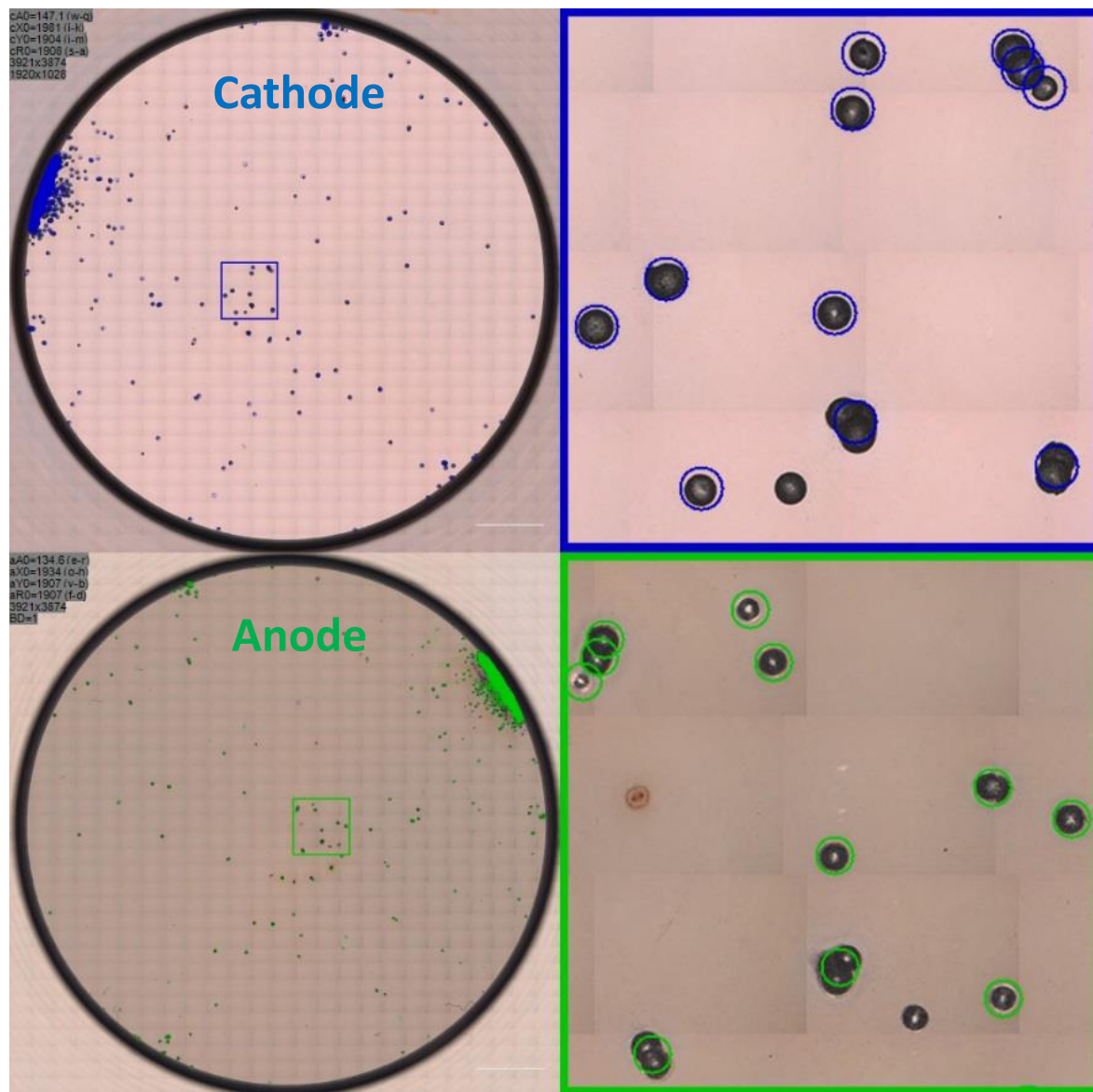


- Image compose of High Quality images





# Large Electrode System (LES)

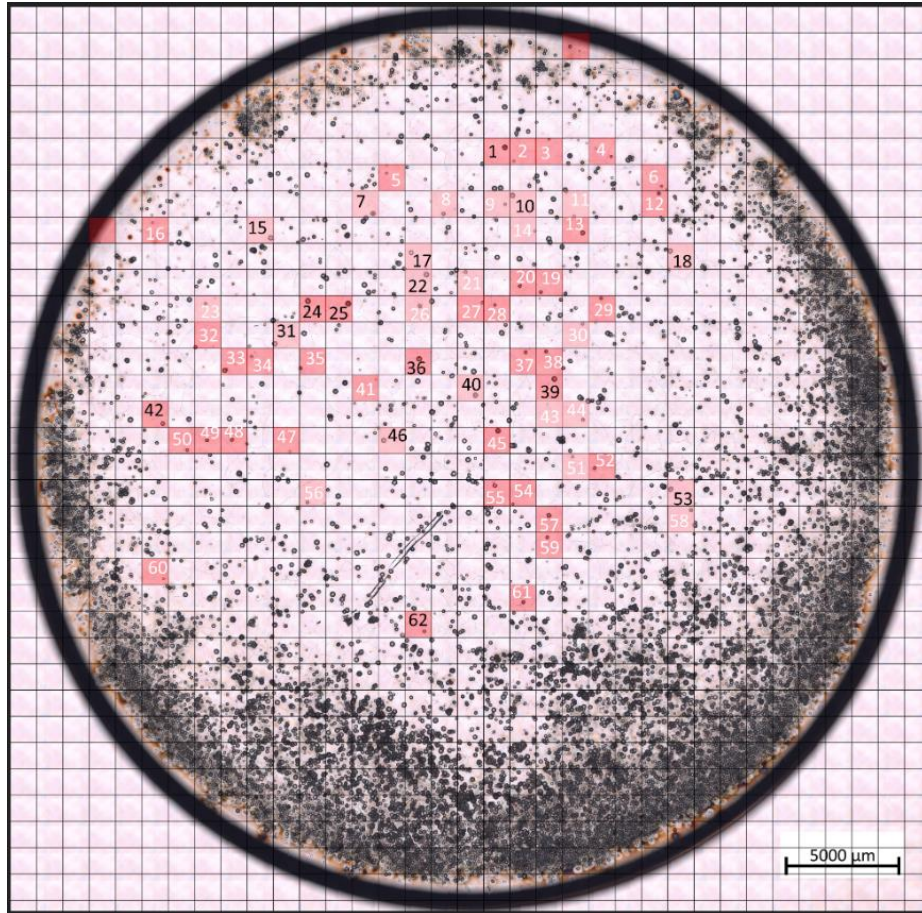


- Combining this techniques we can validate the positioning of the cameras
- Number of breakdowns detected by Marx generator: **5690**
- Number of breakdowns detected by cameras: **5665**
- Difference in data: **~0.5%**.

**Conclusion**  
It works!



# Large Electrode System (LES)



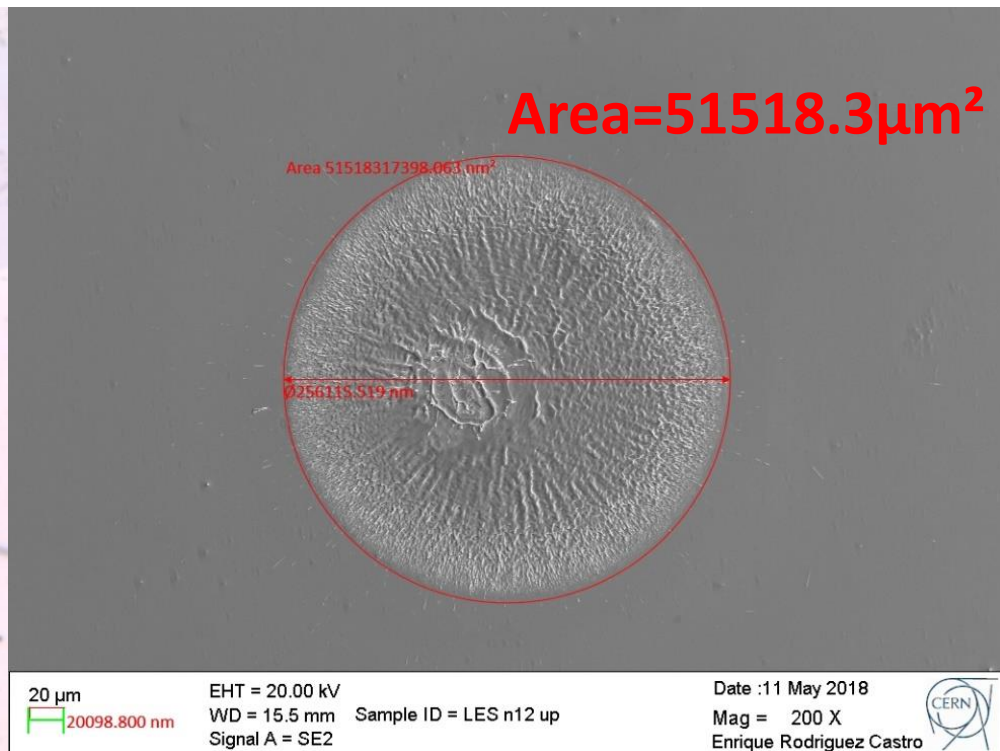
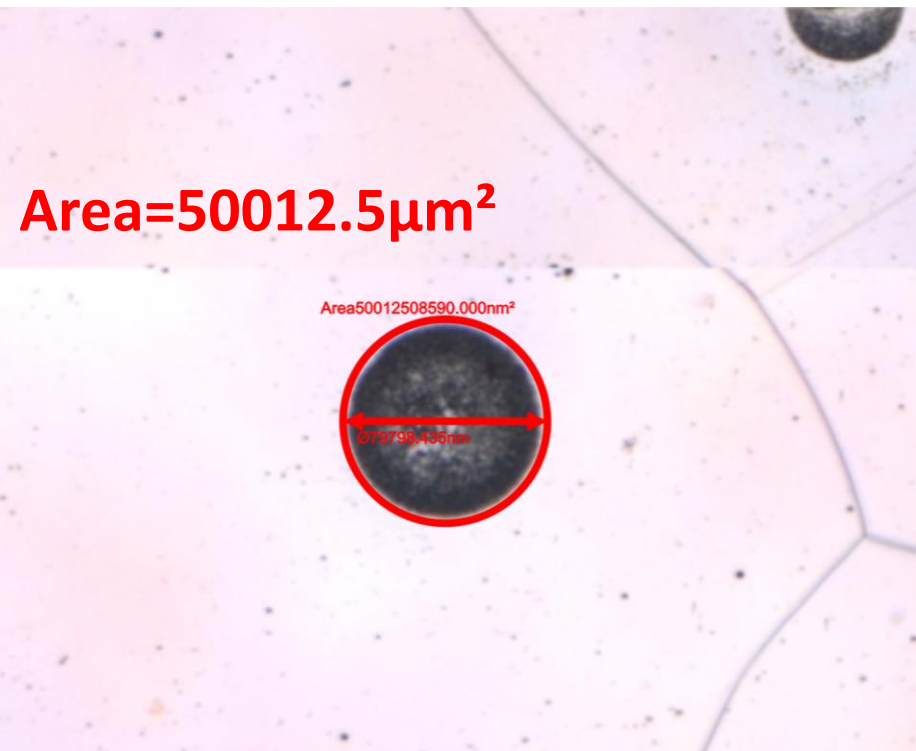
- Is there a relation between the size of the craters and the voltage supplied when they occurred?
- Data analysis:
  - **Cameras** allow to identify single breakdowns locations and voltage
  - **Microscope** to measure the size



# Large Electrode System (LES)



- Measure in OM and SEM
- Average of 60  $\mu\text{m}$  difference in diameter



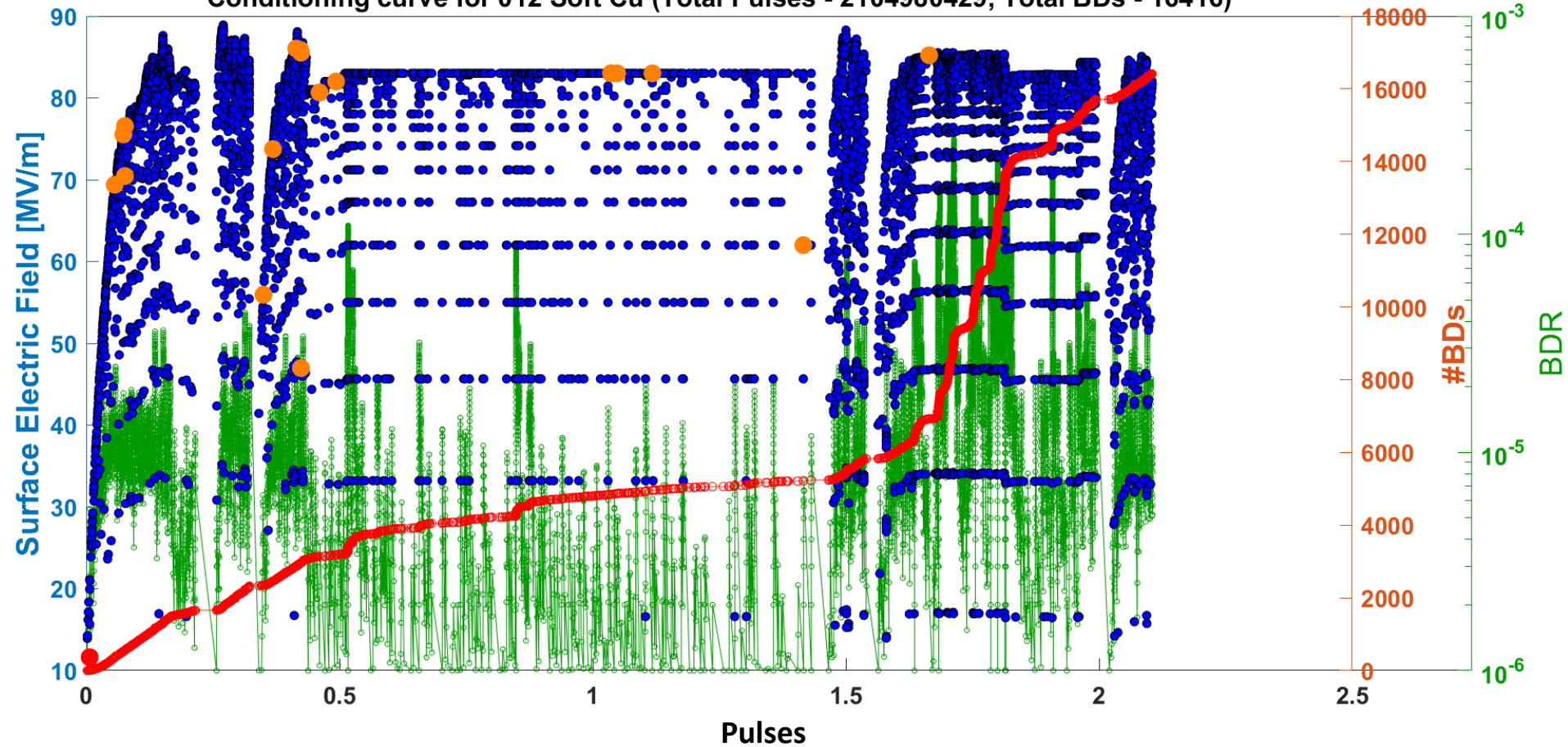
Observations and measurements by Francois-Xavier Greffoz



# Conditioning curve n12



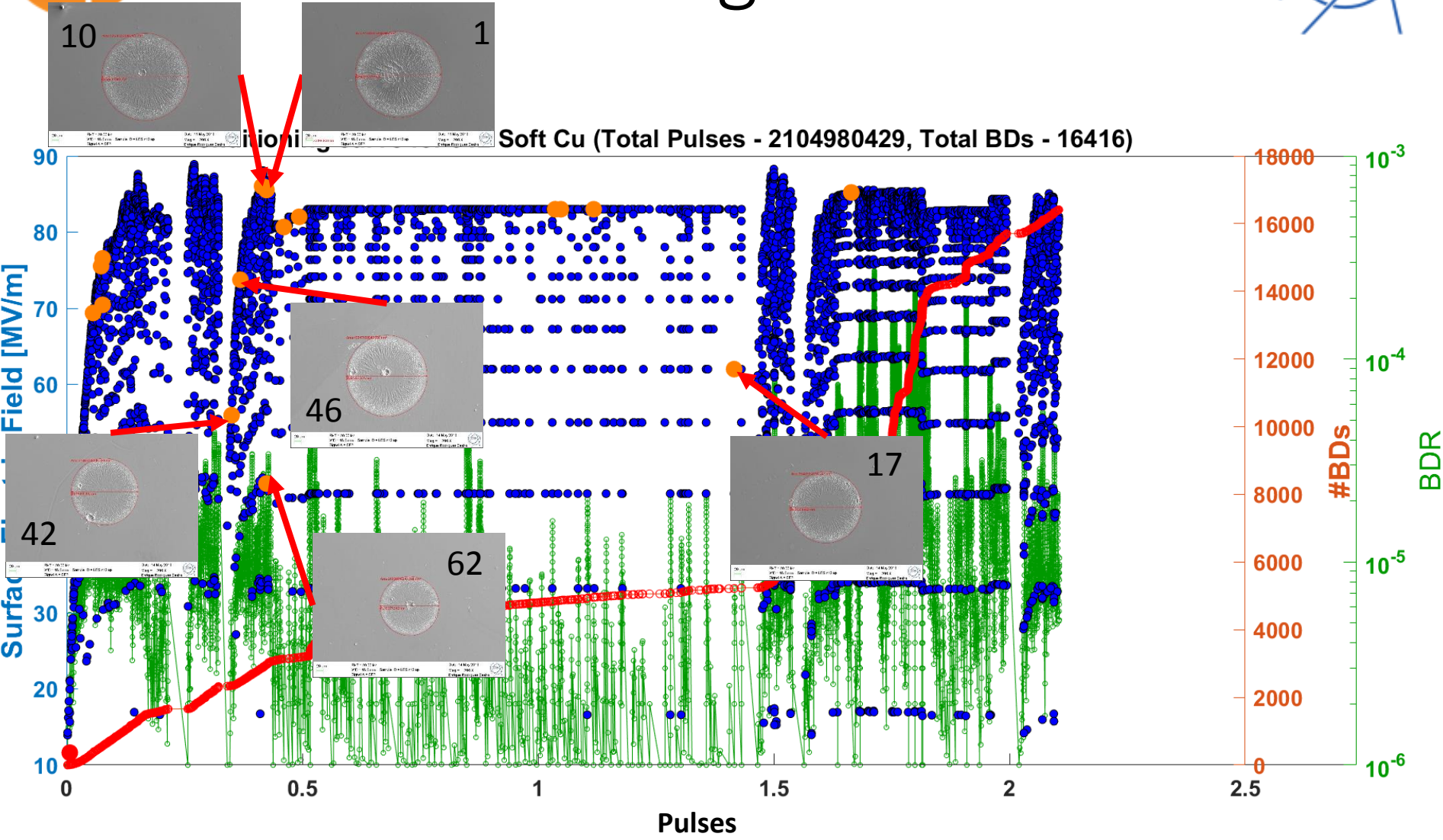
Conditioning curve for 012 Soft Cu (Total Pulses - 2104980429, Total BDs - 16416)





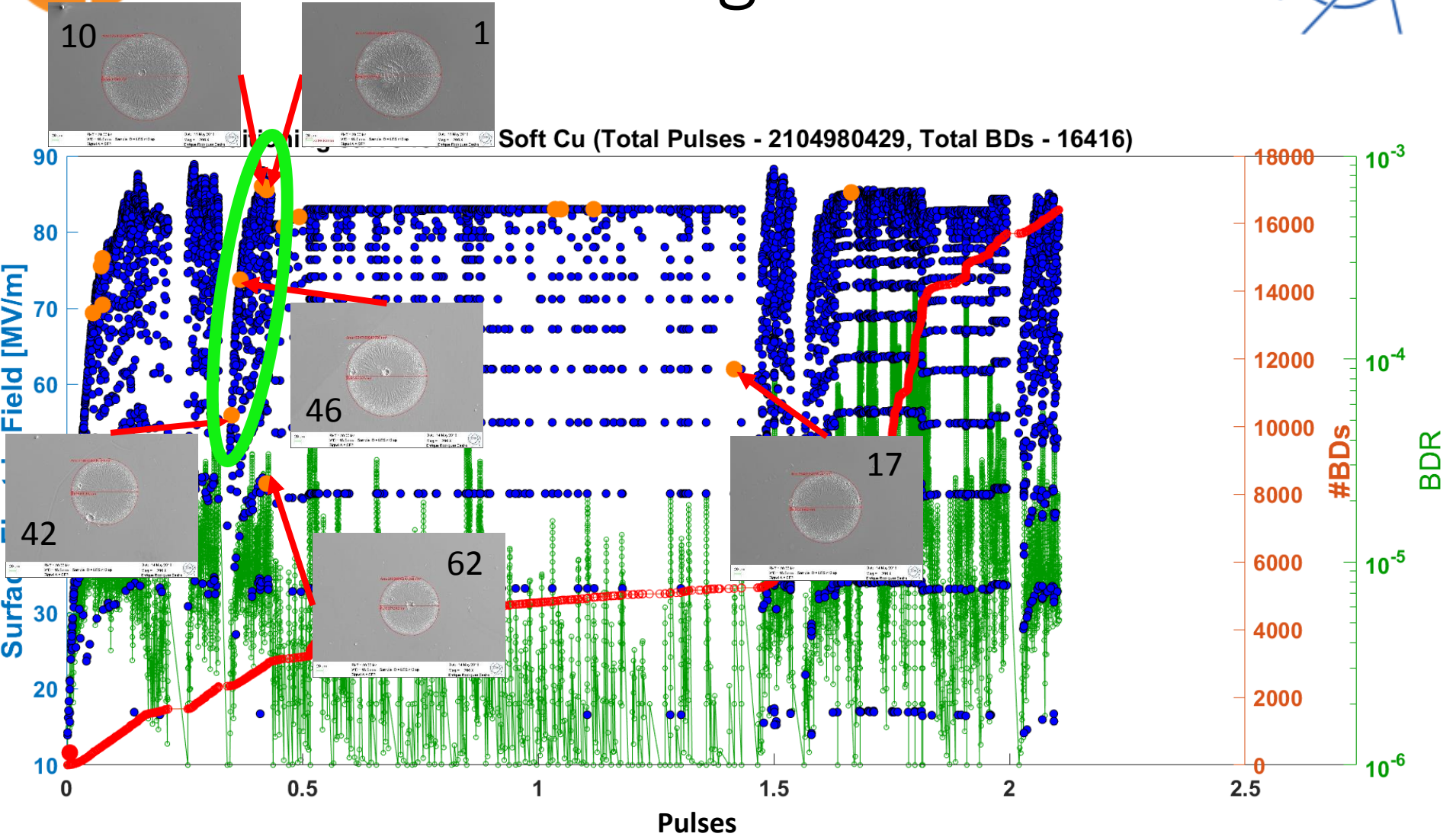


# Conditioning curve n12

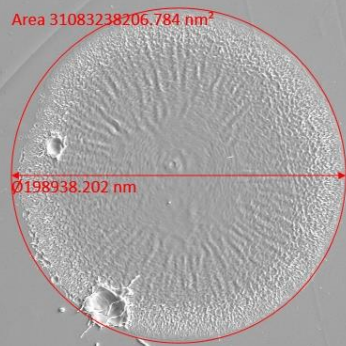




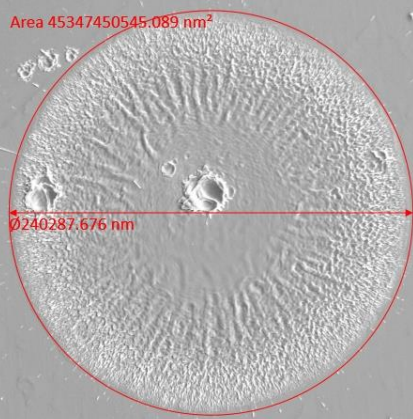
# Conditioning curve n12



42



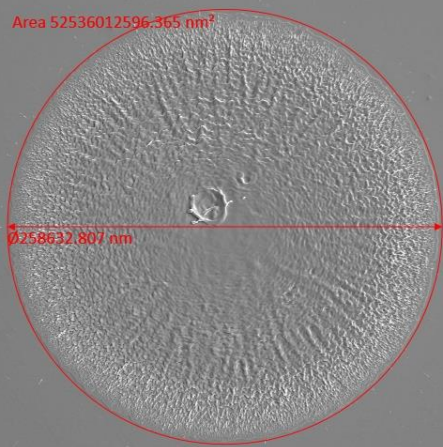
46



20 µm EHT = 20.00 kV Date :14 May 2018   
┌───┐ WD = 15.5 mm Mag = 200 X Enrique Rodriguez Castro  
└───┘ Signal A = SE2 Sample ID = LES n12 up

20 µm EHT = 20.00 kV Date :14 May 2018   
┌───┐ WD = 15.5 mm Mag = 200 X Enrique Rodriguez Castro  
└───┘ Signal A = SE2 Sample ID = LES n12 up

10



1



20 µm EHT = 20.00 kV Date :11 May 2018   
┌───┐ WD = 15.5 mm Mag = 200 X Enrique Rodriguez Castro  
└───┘ Signal A = SE2 Sample ID = LES n12 up

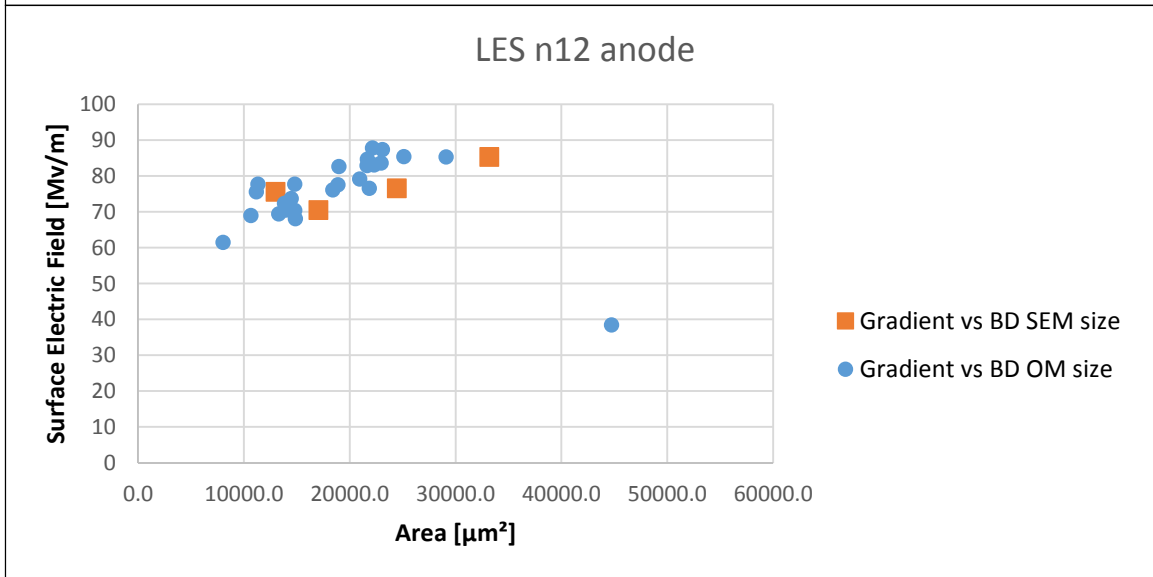
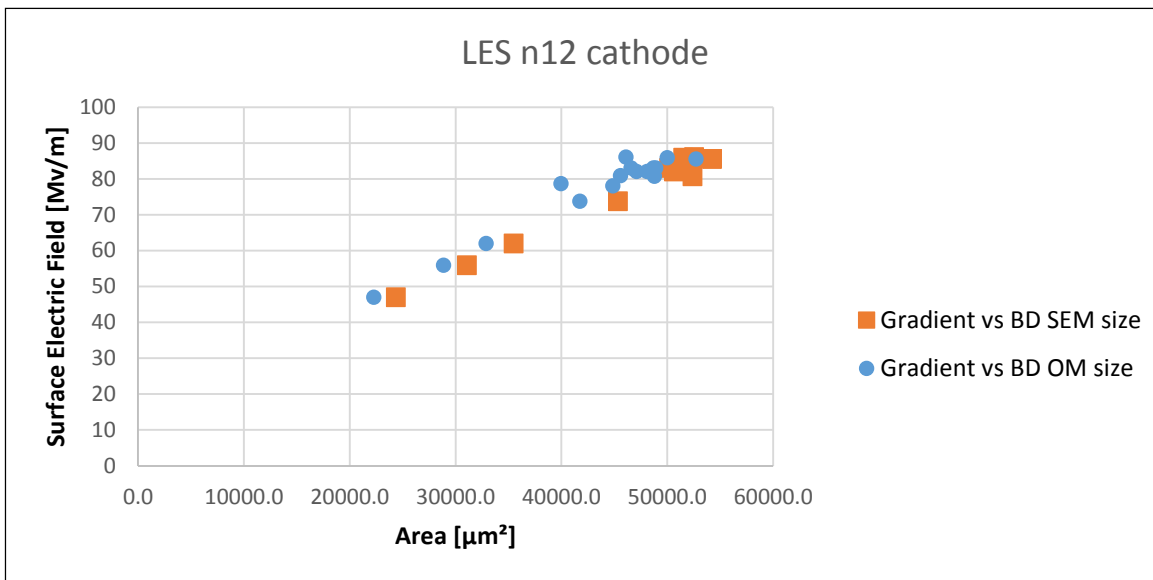
20 µm EHT = 20.00 kV Date :11 May 2018   
┌───┐ WD = 15.5 mm Mag = 200 X Enrique Rodriguez Castro  
└───┘ Signal A = SE2 Sample ID = LES n12 up



# Large Electrode System (LES)



- Preliminary results show that crater size increase with surface electric field

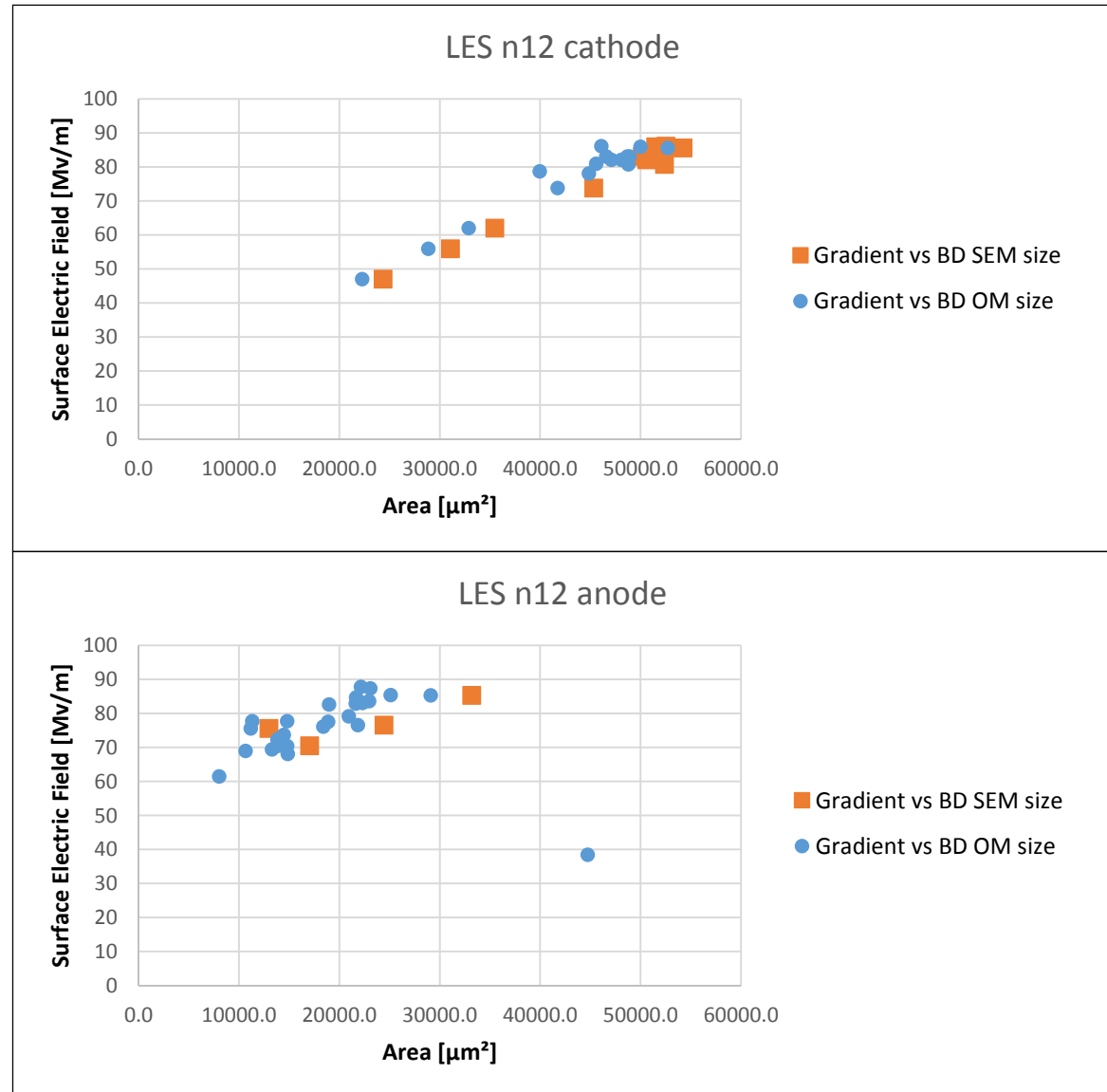




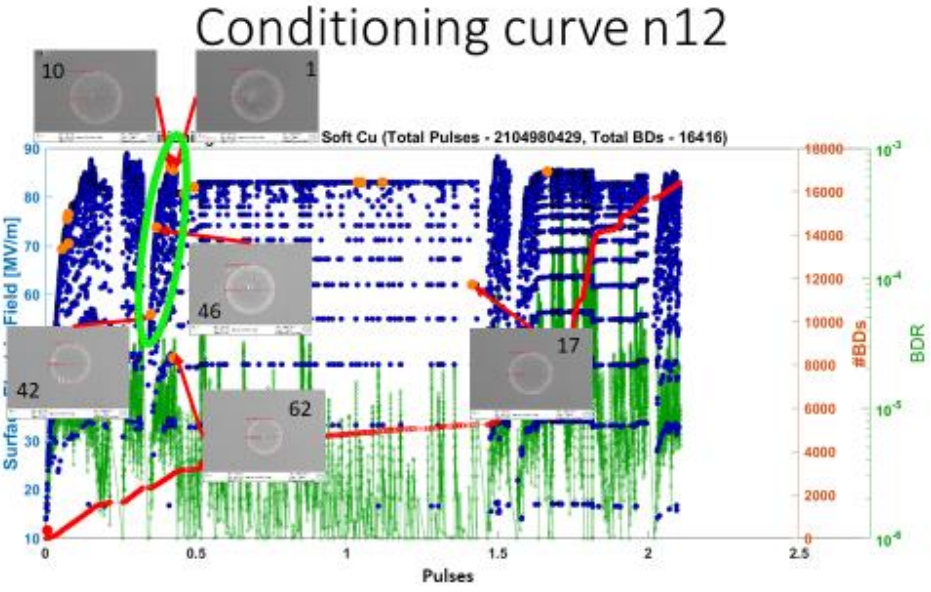
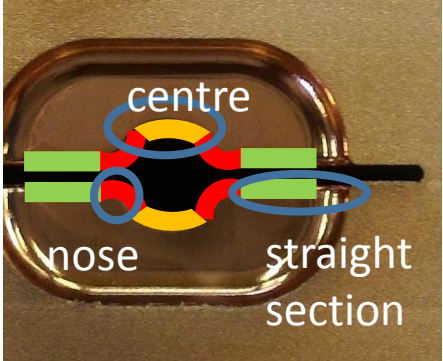
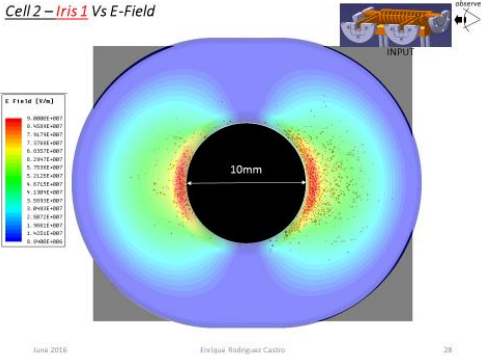
# Large Electrode System (LES)



- Preliminary results show that crater size increase with surface electric field



- Post mortem on RF and DC important tool on BD understanding
- Constantly adding equipment and tools
  - FIB-SEM → cross section and lamellas
  - APA → pollution analysis
  - Cameras → BD positioning not only in space but also in time
- CLIC-AS: BD accumulation on max E<sub>field</sub> regions
- LES: BD size relation to surface electric field



# Thank you for you attention

Questions?

100  $\mu$ m



EHT = 20.00 kV  
WD = 10.0 mm  
Signal A = SE2

Sample ID = G02-1\_

Stage at T = 0.0 °

Enrique Rodriguez Castro  
Date :7 Nov 2016  
Mag = 200 X





# Recent microscopy at CERN

*Enrique Rodríguez Castro*

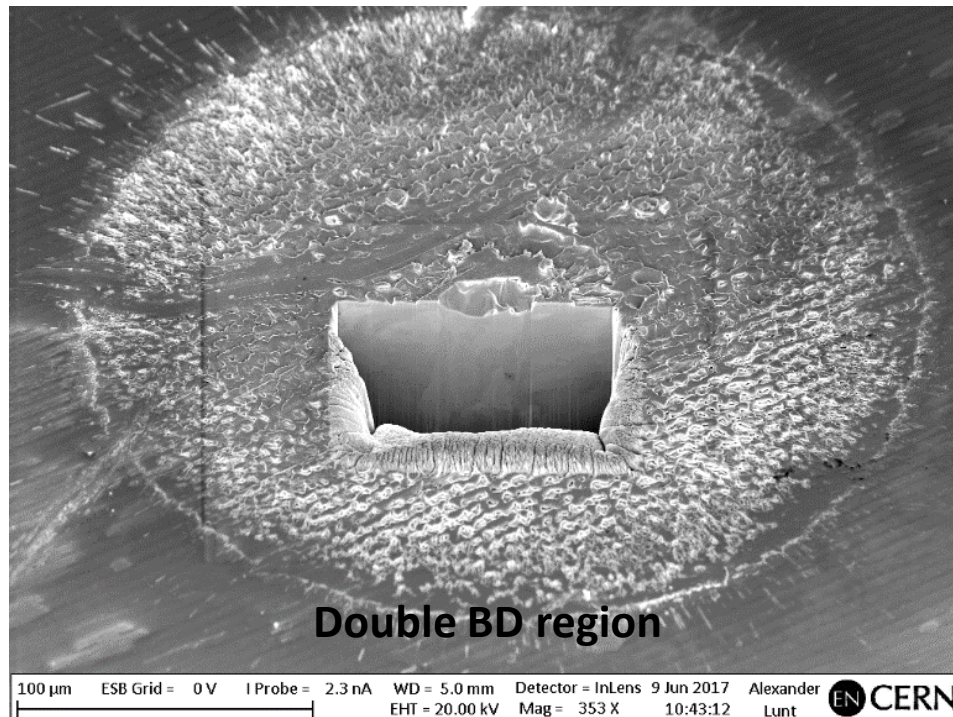
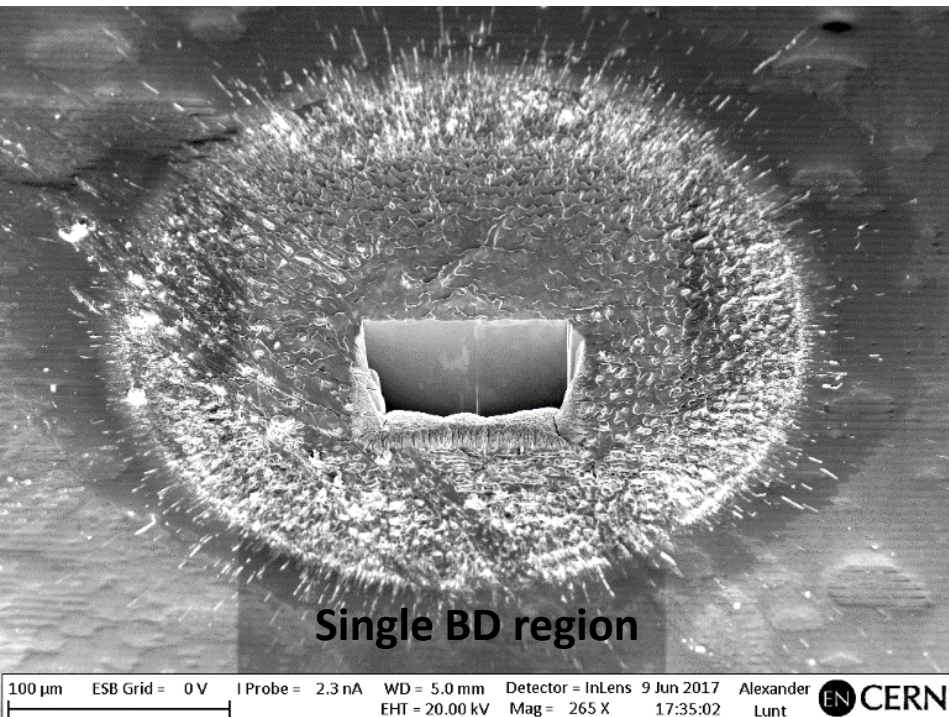






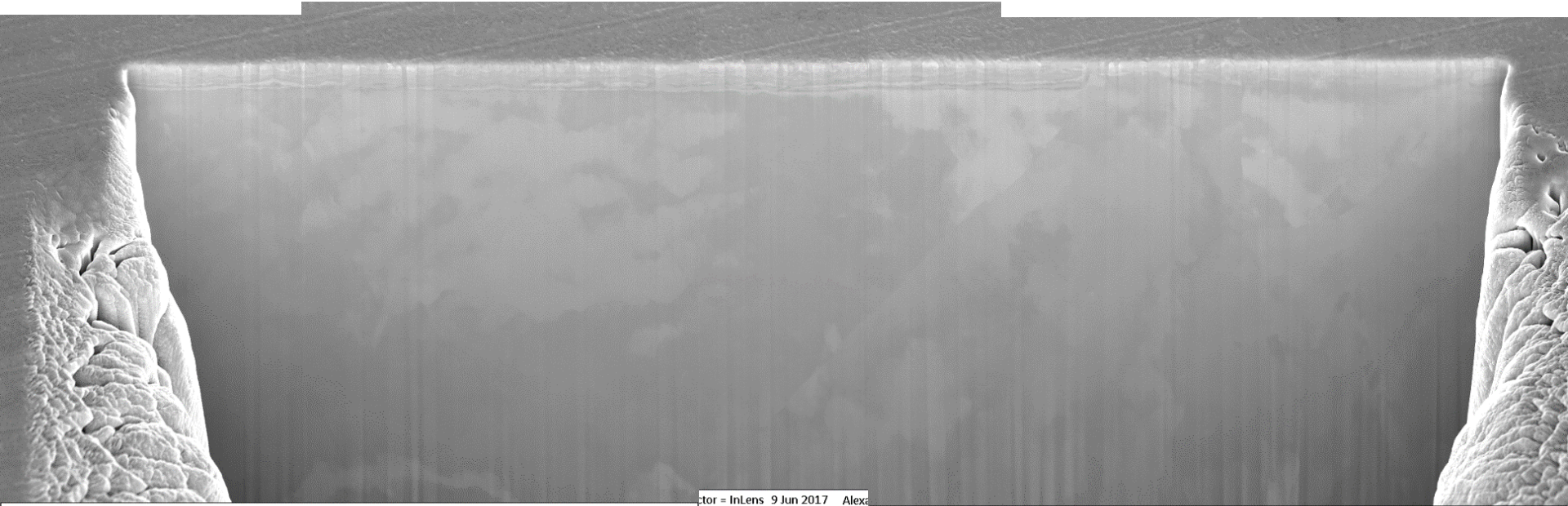
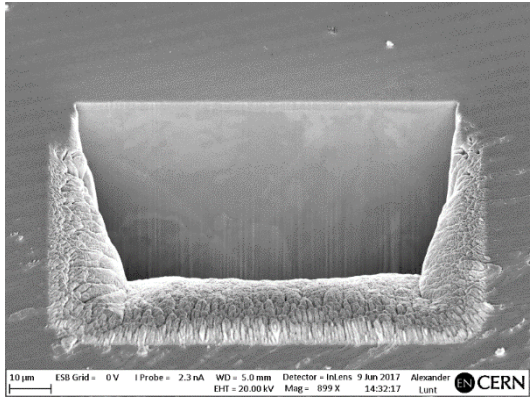
# EXTRA SLIDES

- 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



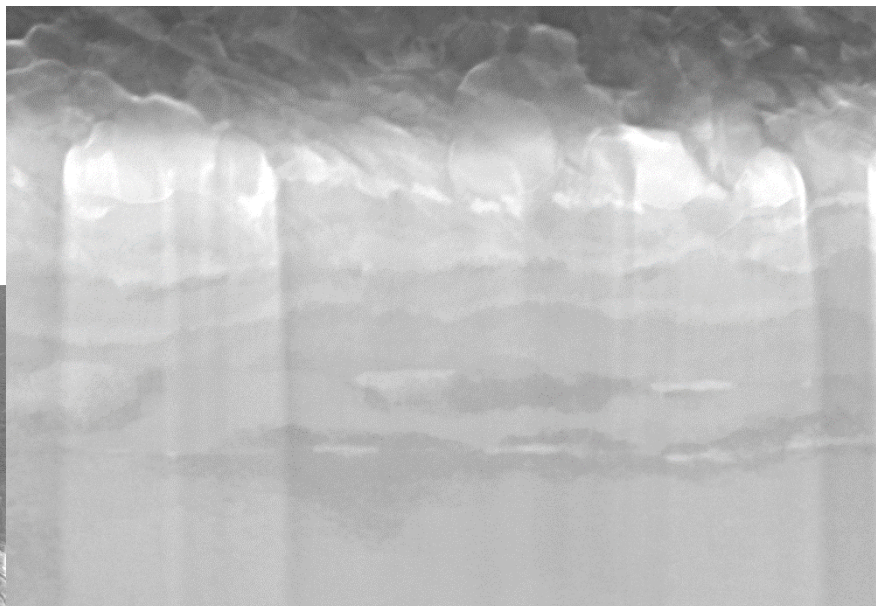
10  $\mu$ m ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN  
EHT = 20.00 kV Mag = 2.44 K X 14:34:38

Detector = InLens 9 Jun 2017 Alexander Lunt  
Mag = 2.44 K X 14:34:00

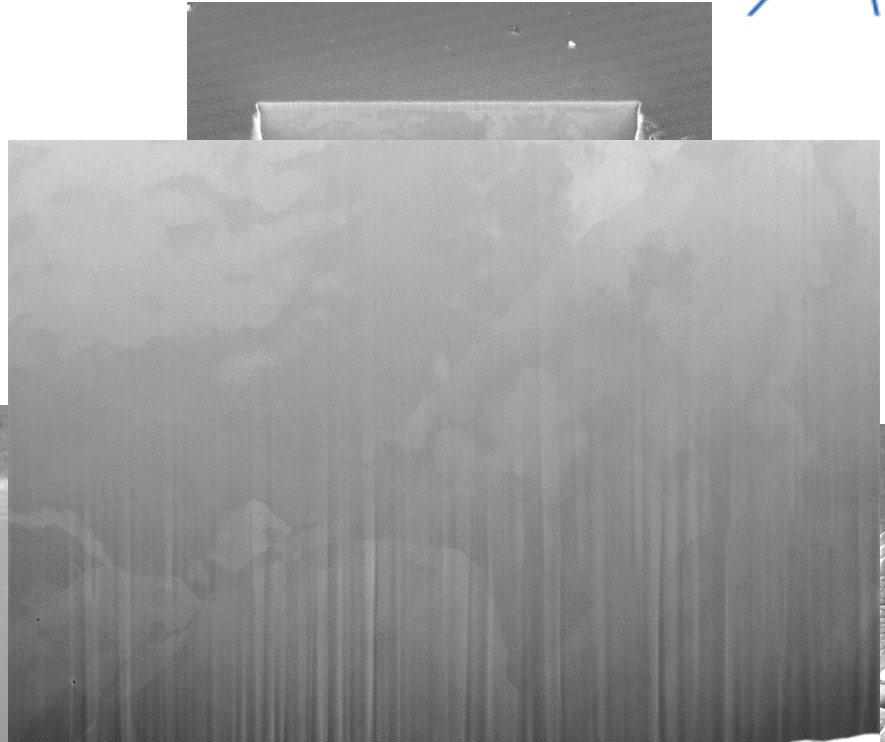
10  $\mu$ m ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN  
EHT = 20.00 kV Mag = 2.44 K X 14:33:30



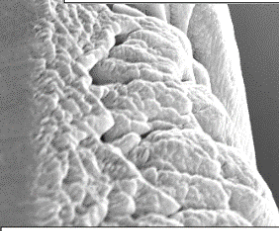
# Cross-section: Hard Cu Reference



1  $\mu$ m ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN  
EHT = 20.00 kV Mag = 24.69 K X 14:37:06



10  $\mu$ m ESB Grid = 0 V I Probe = 2.3 nA WD = 5.1 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN  
EHT = 20.00 kV Mag = 2.10 K X 14:35:13



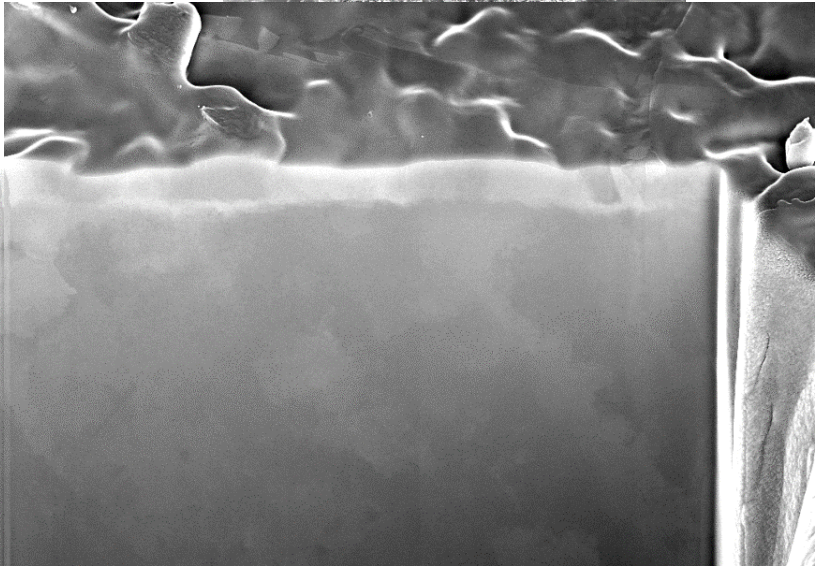
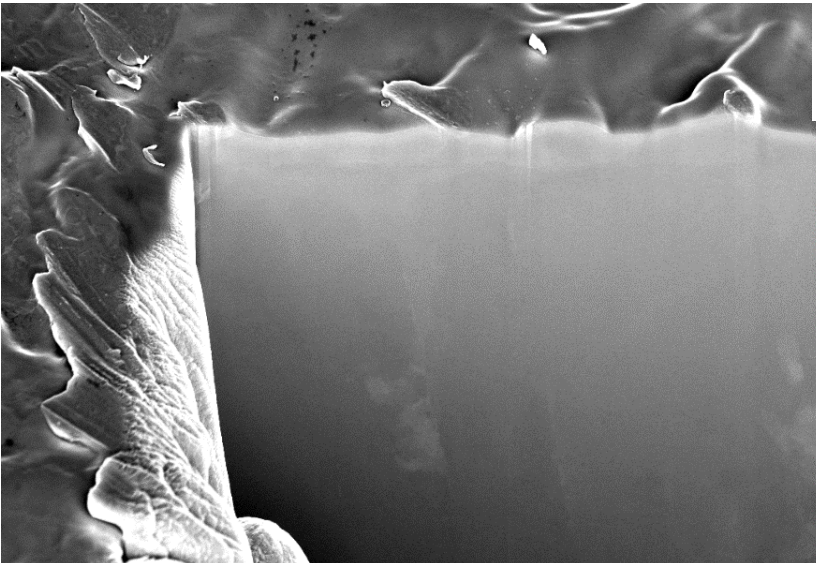
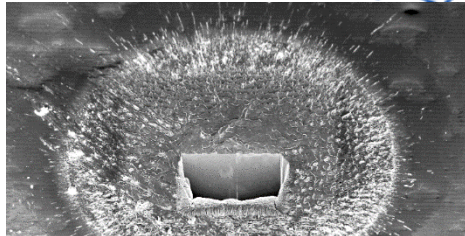
10  $\mu$ m ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN  
EHT = 20.00 kV Mag = 2.44 K X 14:34:38

Detector = InLens 9 Jun 2017 Alexander Lunt  
Mag = 2.44 K X 14:34:00

10  $\mu$ m ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN  
EHT = 20.00 kV Mag = 2.44 K X 14:33:30



# Cross-section: Hard Cu 1 BD



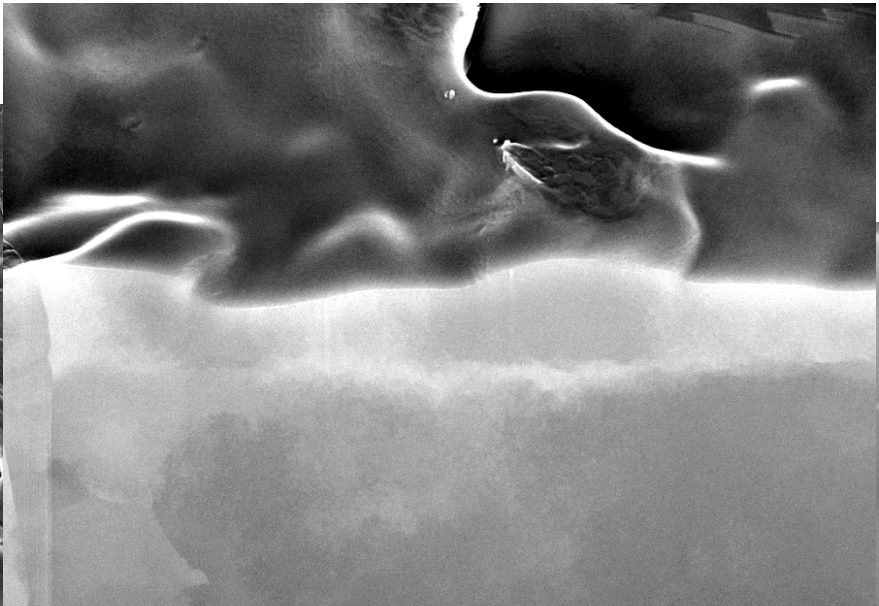
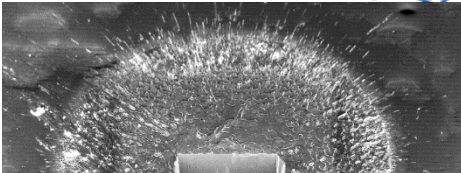
10 µm ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt CERN  
EHT = 20.00 kV Mag = 2.47 K X 17:37:25

10 µm ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt CERN  
EHT = 20.00 kV Mag = 2.47 K X 17:36:51

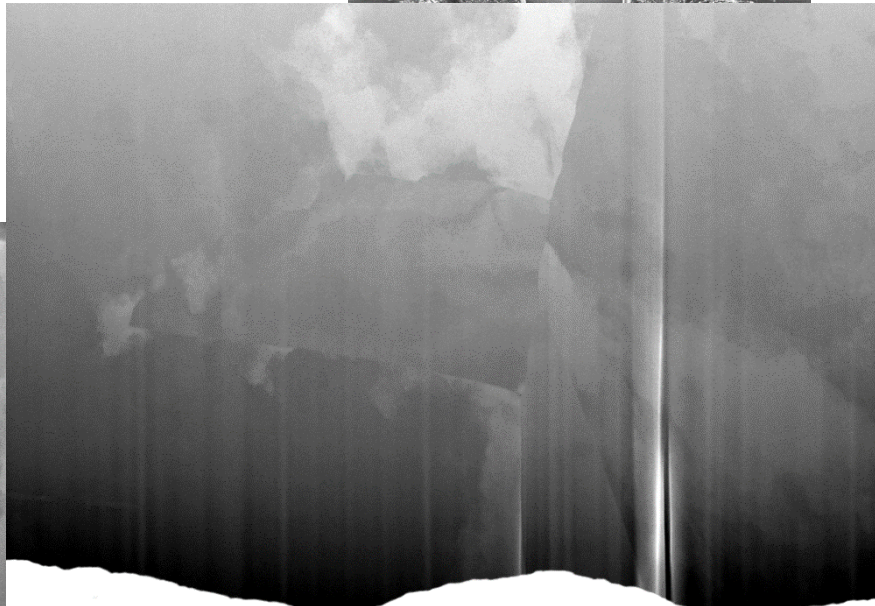
10 µm ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt CERN  
EHT = 20.00 kV Mag = 2.47 K X 17:37:09



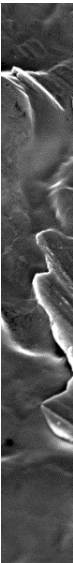
# Cross-section: Hard Cu 1 BD



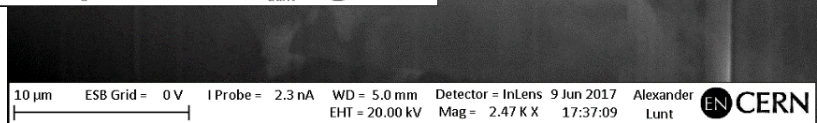
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EHT = 20.00 kV Mag = 5.43 K X 17:39:47 Lunt



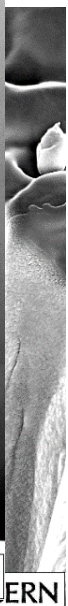
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EHT = 20.00 kV Mag = 2.77 K X 17:38:13 Lunt



10 µm ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander EN CERN  
EHT = 20.00 kV Mag = 2.47 K X 17:37:25 Lunt



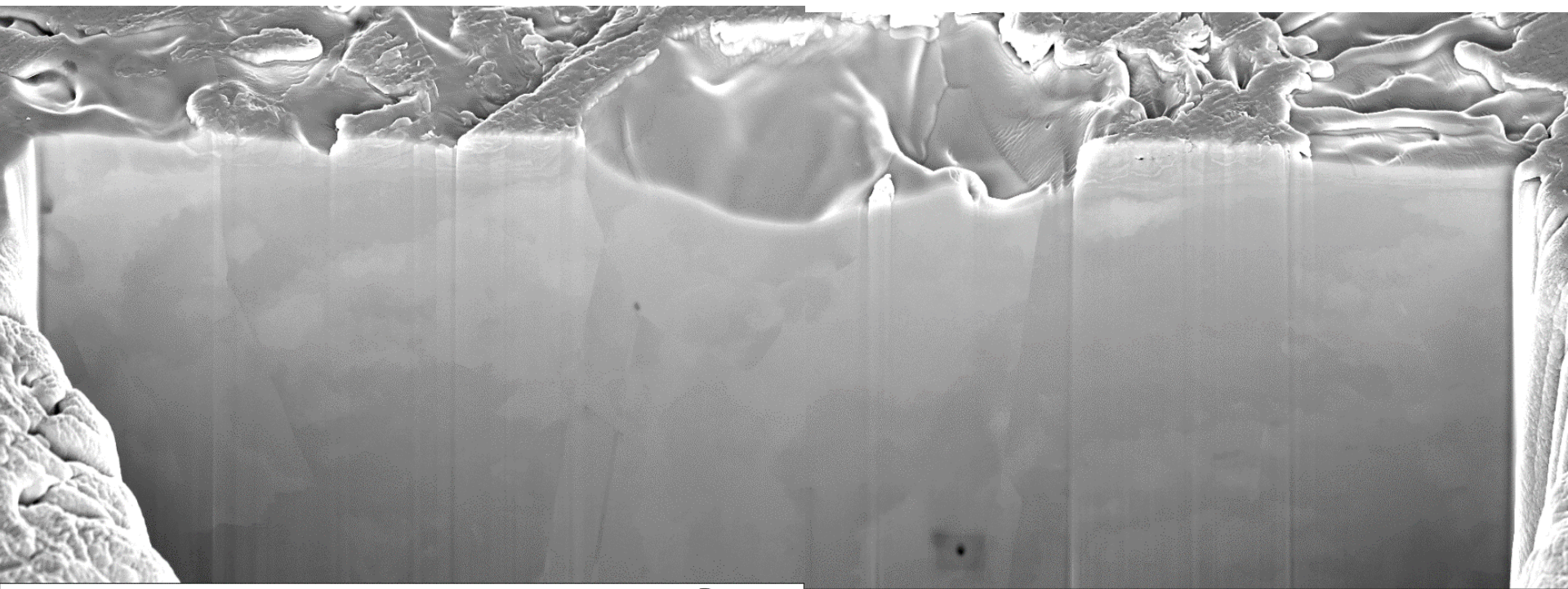
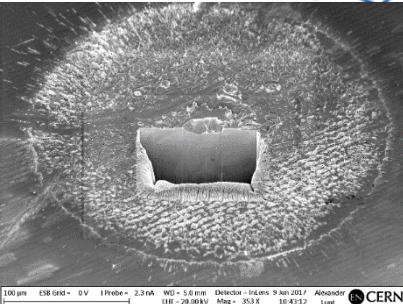
10 µm ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander EN CERN  
EHT = 20.00 kV Mag = 2.47 K X 17:37:09 Lunt



10 µm ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander EN CERN  
EHT = 20.00 kV Mag = 2.47 K X 17:36:51 Lunt



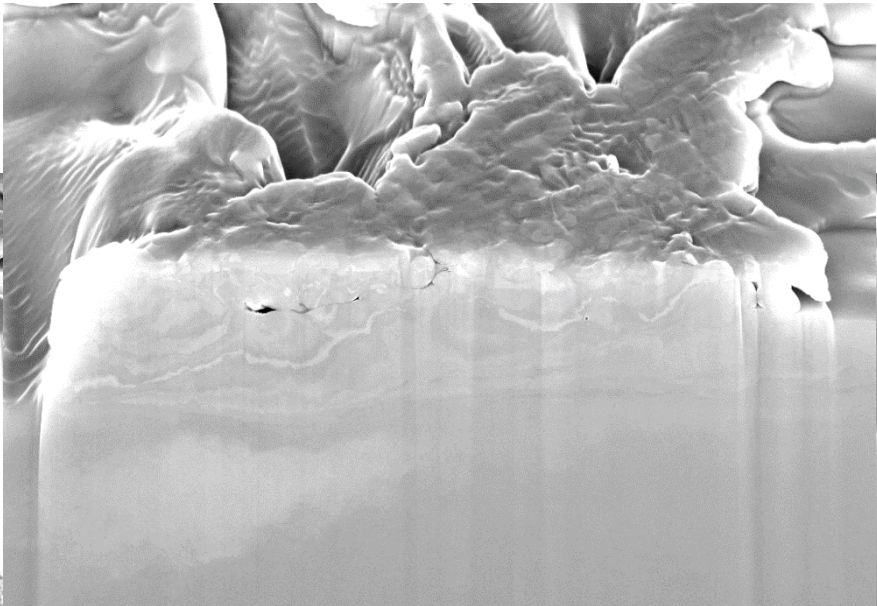
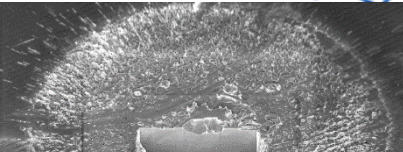
# Cross-section: Hard Cu 2 BD



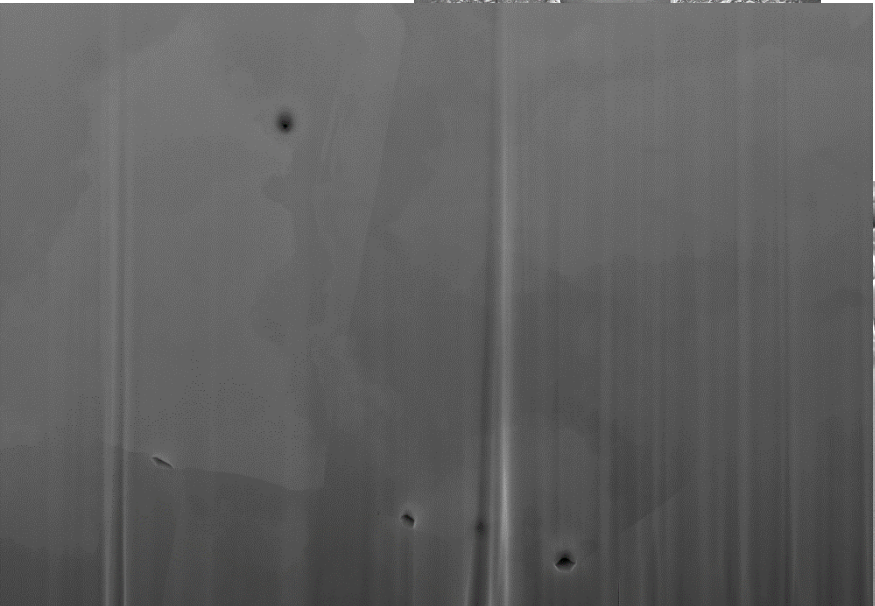
10 μm ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN  
EHT = 20.00 kV Mag = 2.26 K X 10:40:47 EHT = 20.00 kV Mag = 2.26 K X 10:41:44



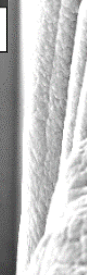
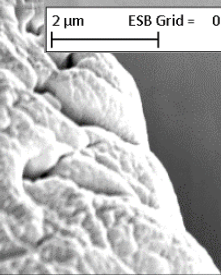
# Cross-section: Hard Cu 2 BD



2 µm ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN  
EHT = 20.00 kV Mag = 6.96 K X 10:45:59



10 µm ESB Grid = 0 V I Probe = 2.3 nA WD = 5.1 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN  
EHT = 20.00 kV Mag = 4.10 K X 10:38:04



10 µm ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN  
EHT = 20.00 kV Mag = 2.26 K X 10:40:47

ESB Grid = 0 V I Probe = 2.3 nA WD = 5.0 mm Detector = InLens 9 Jun 2017 Alexander Lunt EN CERN  
EHT = 20.00 kV Mag = 2.26 K X 10:41:44