Structural Features of Hard Cu Electrodes Tested at 300 and 30 K

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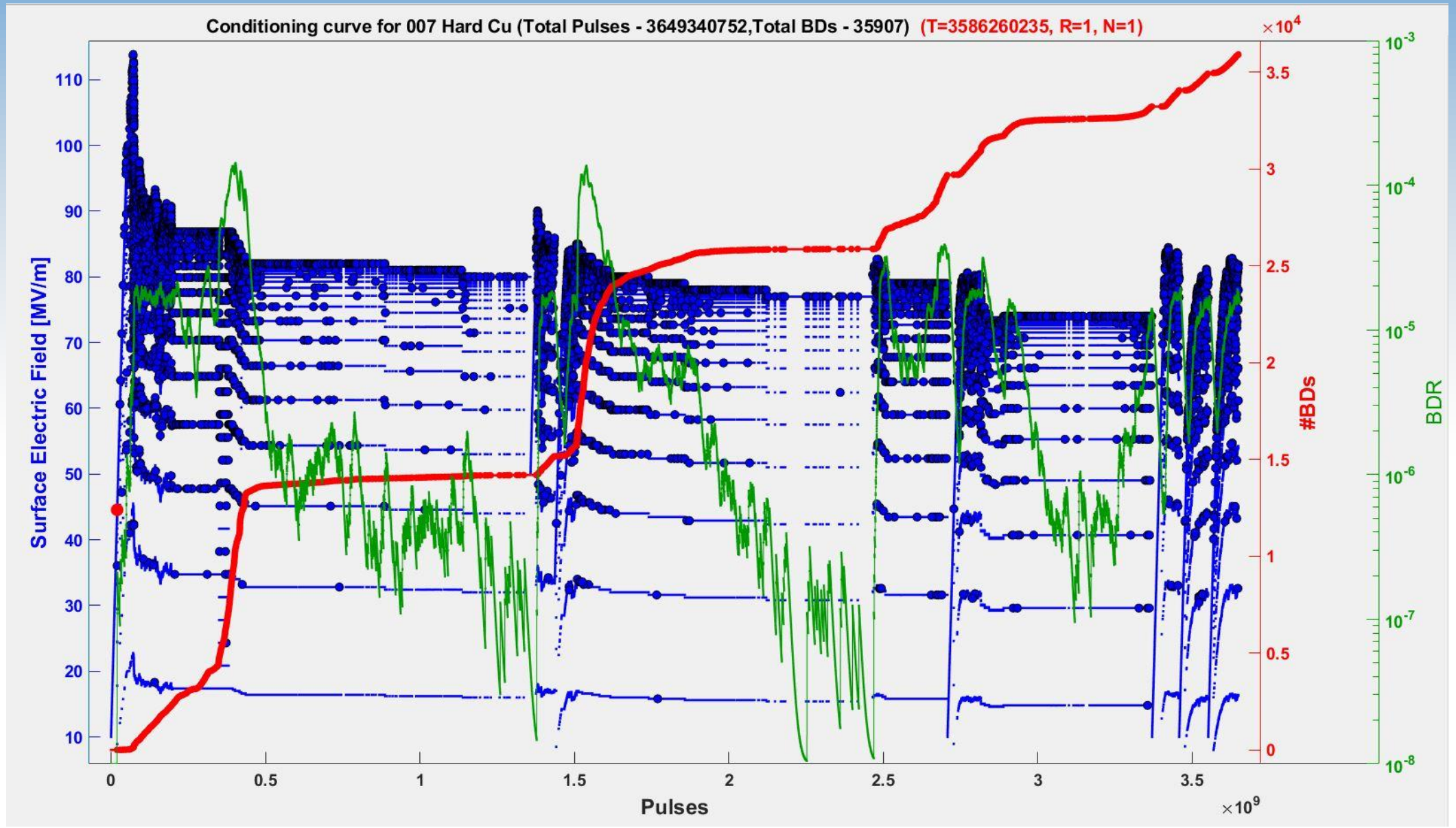
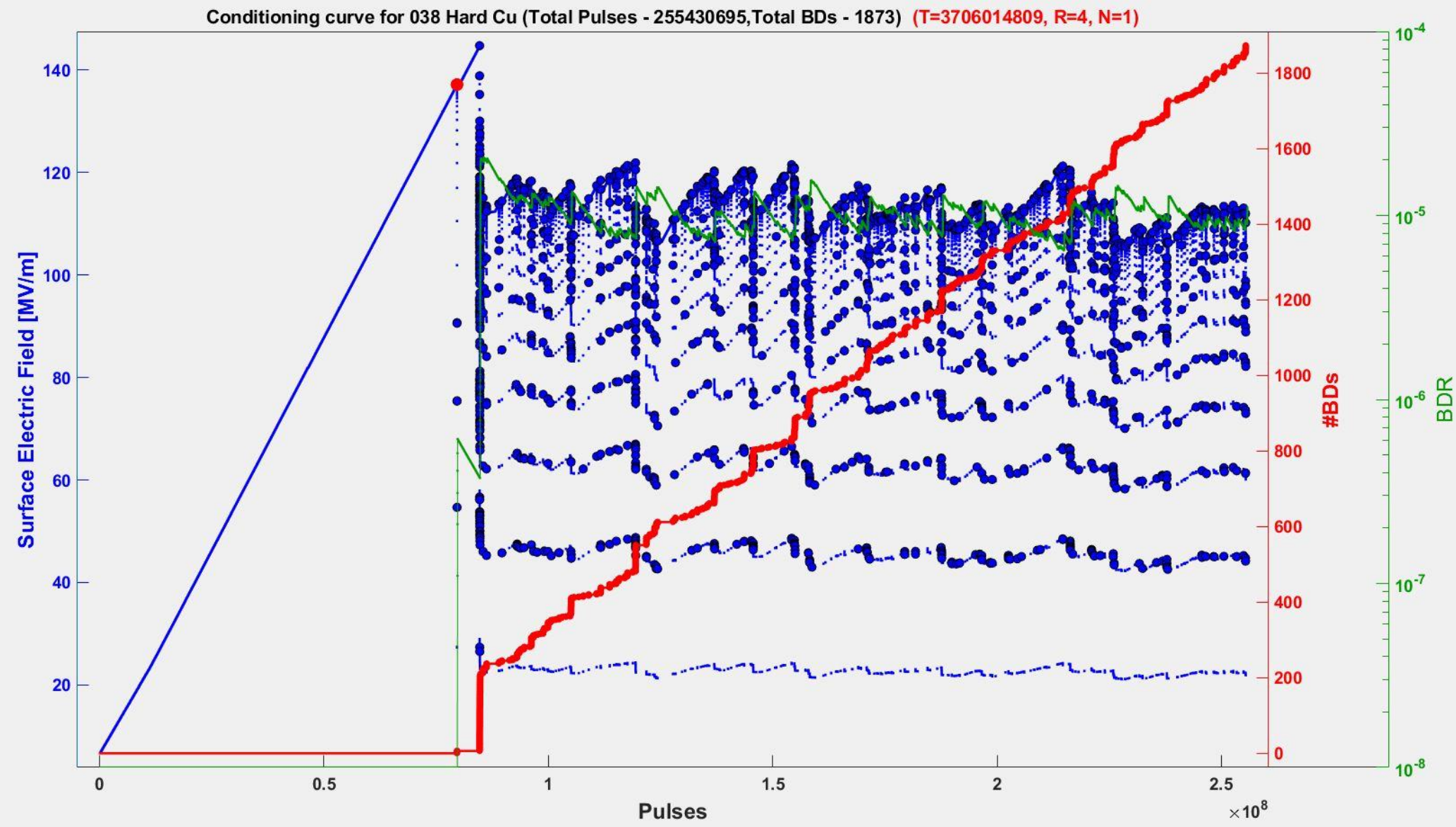
OUR STARTING POINTS:

No.	Parameter	007 Hard Cu	038 Hard Cu
1.	Specification for Cu	OFE-Cu	OFE-Cu
2.	Manufactured by	Ets Yvon BOYER S.A.S.	VDL ETG
3.	Cleaning procedure for electrodes	EDMS 1390360 x 2 times + passivation with chromic acid (because of oxidation)	EDMS 1390360
4.	Cathode	Hard Cu № 186-06 (40 mm diameter)	Hard Cu № 185-12 (40 mm diameter)
5.	Anode	Hard Cu № 186-07 (40 mm diameter)	Hard Cu № 186-13 (60 mm diameter)
6.	Information about the gap between electrodes	~60 µm gap	~59 µm gap (at cold)
7.	Vacuum chamber	LES 2, CERN	Cryo DC system, Uppsala
8.	Temperature	300 K	30 K
9.	Total number of breakdown/pulses	35907/3.6E+9	1873/2.6E+8
10.	E field, MV/m	114.5 MV/m (max) 83 MV/m (average in the end)	144.6 MV/m (max) 122 MV/m (average in the end)

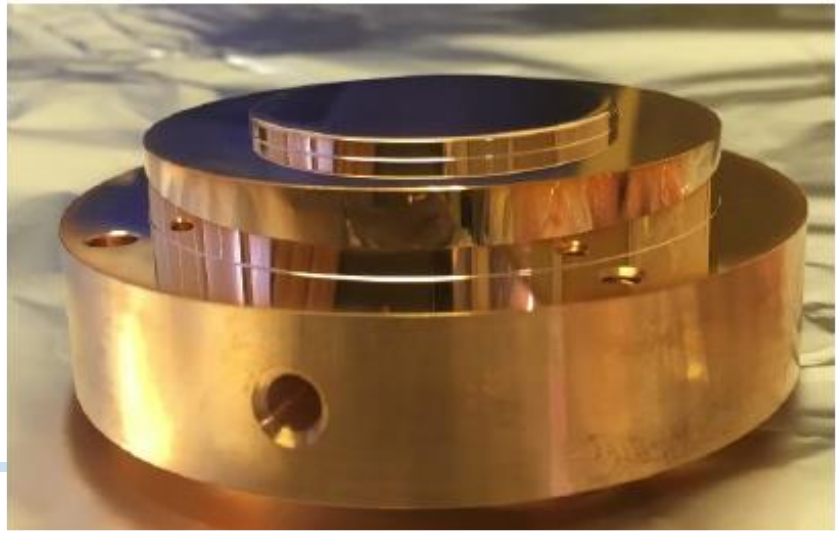
Hard Cu Electrode Pair 007 tested at 300 K: Full Test History

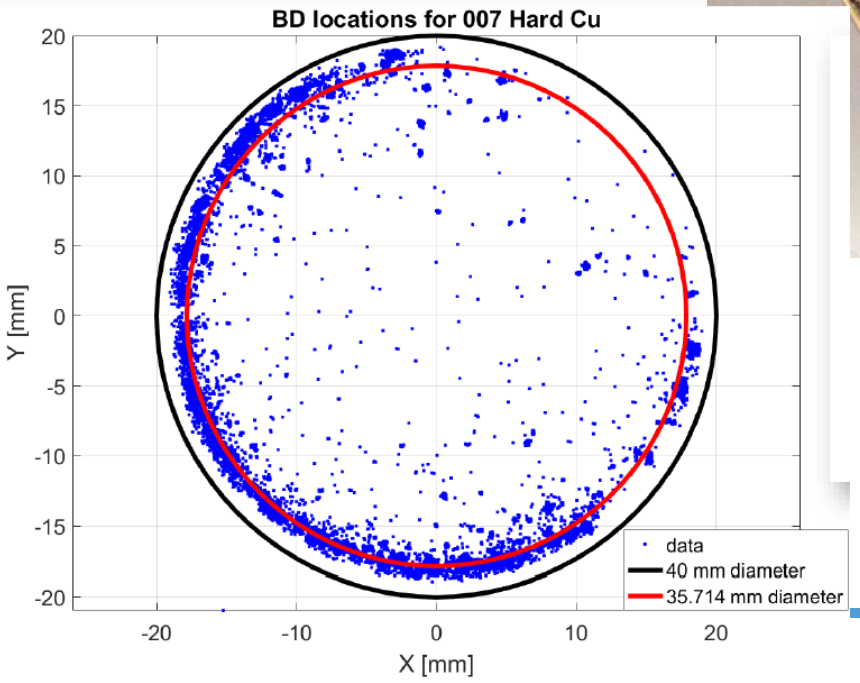


Hard Cu Electrode Pair 038 tested at 30 K: Full Test History



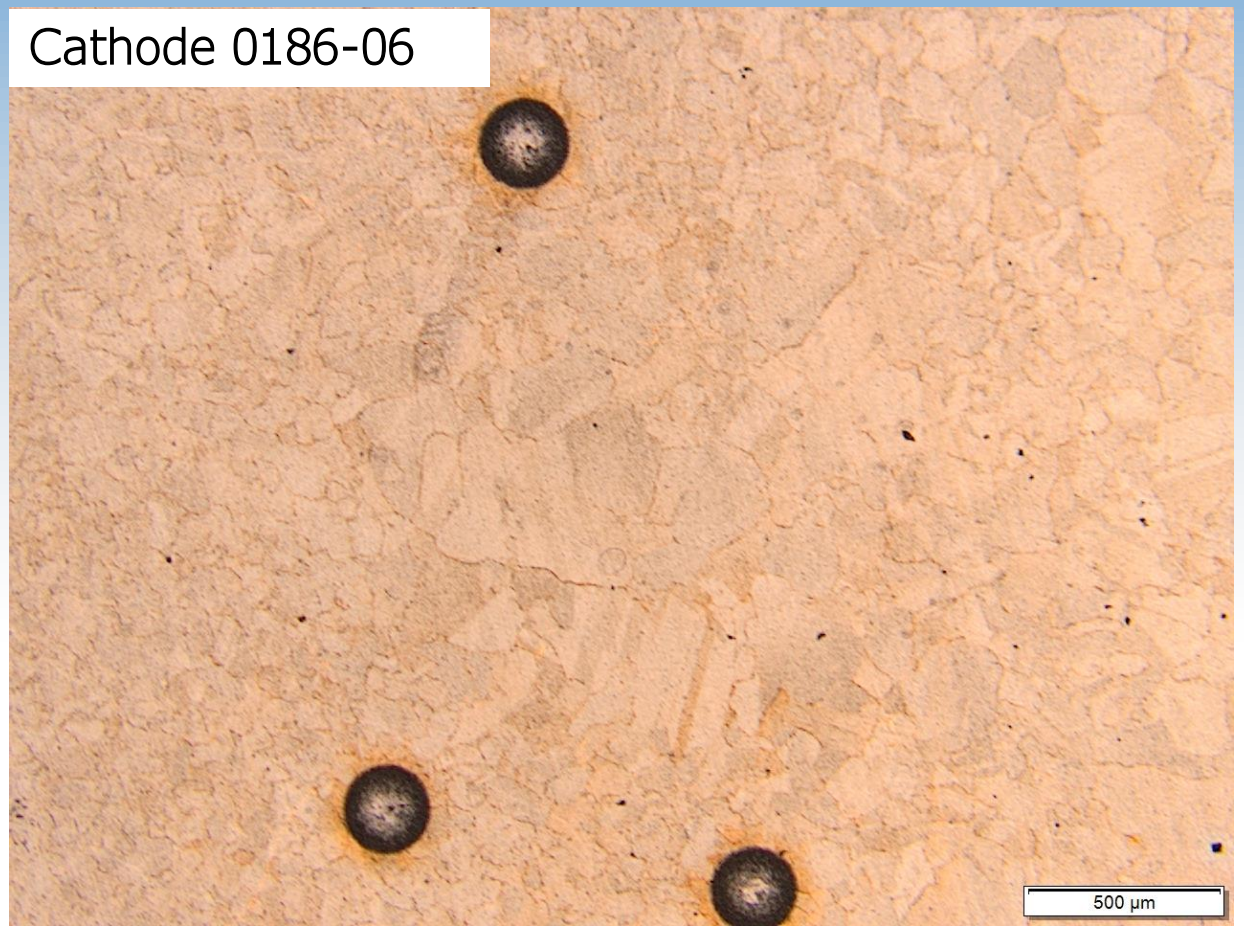
Hard Cu Electrode Pair 007 tested at 300 K: BD localization



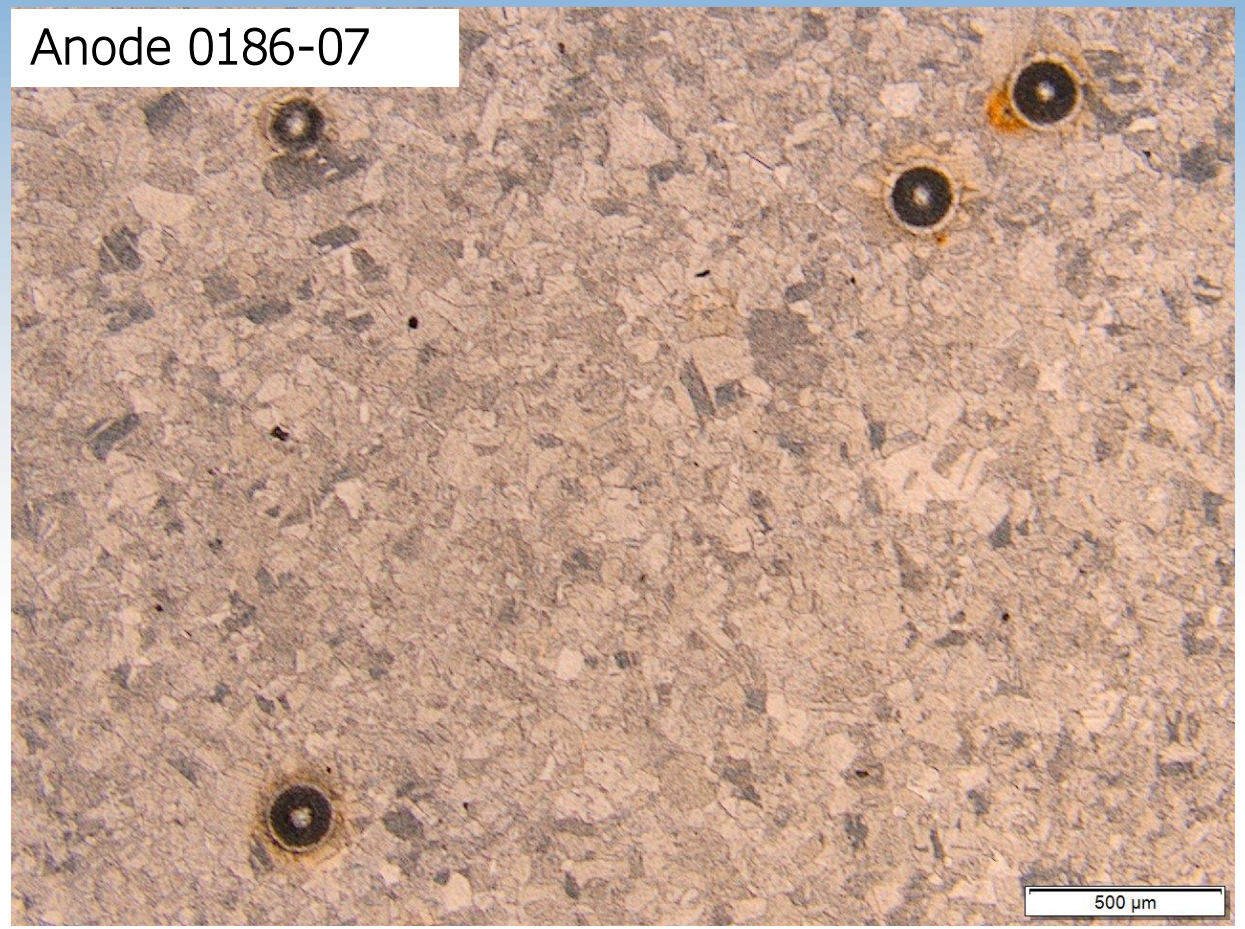


Hard Cu Electrode Pair 007 tested at 300 K: surface in LM

Cathode 0186-06



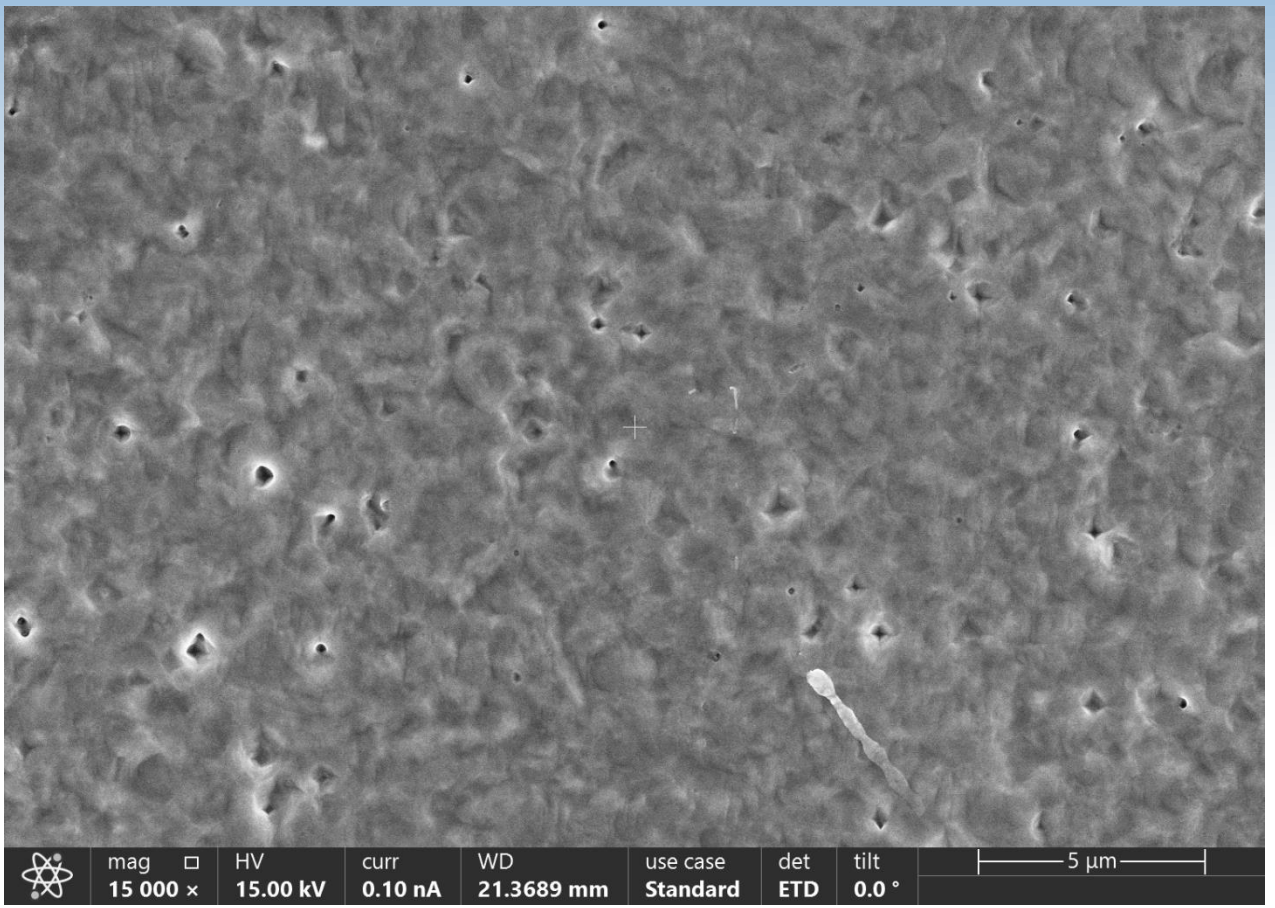
Anode 0186-07

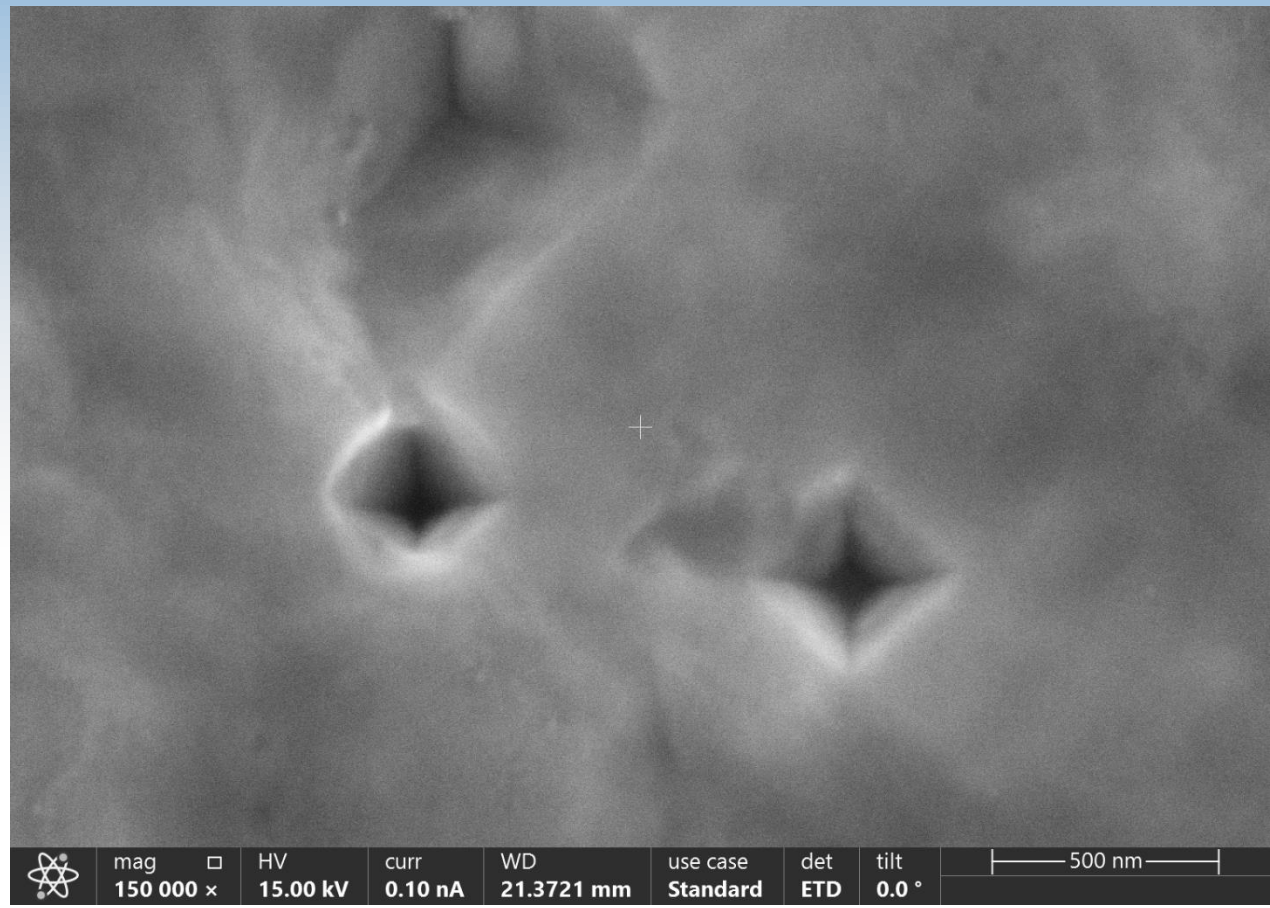


1. The grains are in the range of 20-80 µm out of the central part, 100-400 µm in the center
2. GBs are clearly visible everywhere. The surface is mate(= etched)
3. BDs are localized at the circumference of electrodes
4. No star-like BDs

Hard Cu Electrode Pair 007 tested at 300 K: surface in SEM

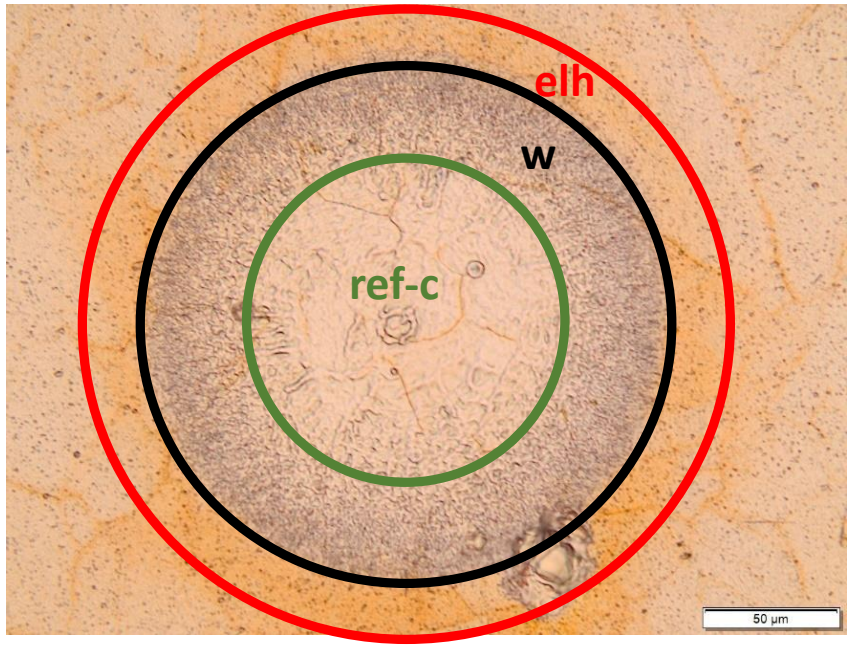
Cathode, in-between BDs





The surface is etched, ~ 250 nm pits are clearly visible

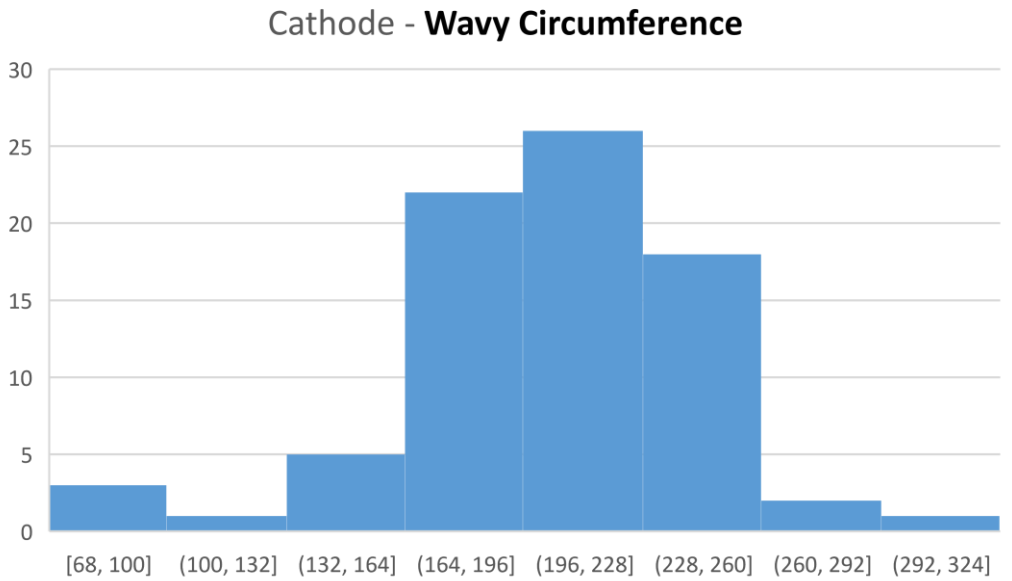
Hard Cu cathode 0186-06 tested at 300 K: morphology@sizing of a typical BD

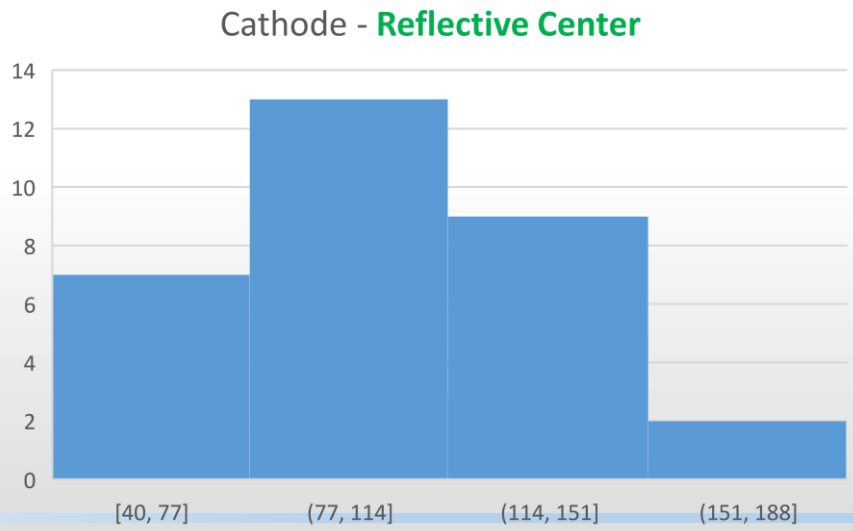


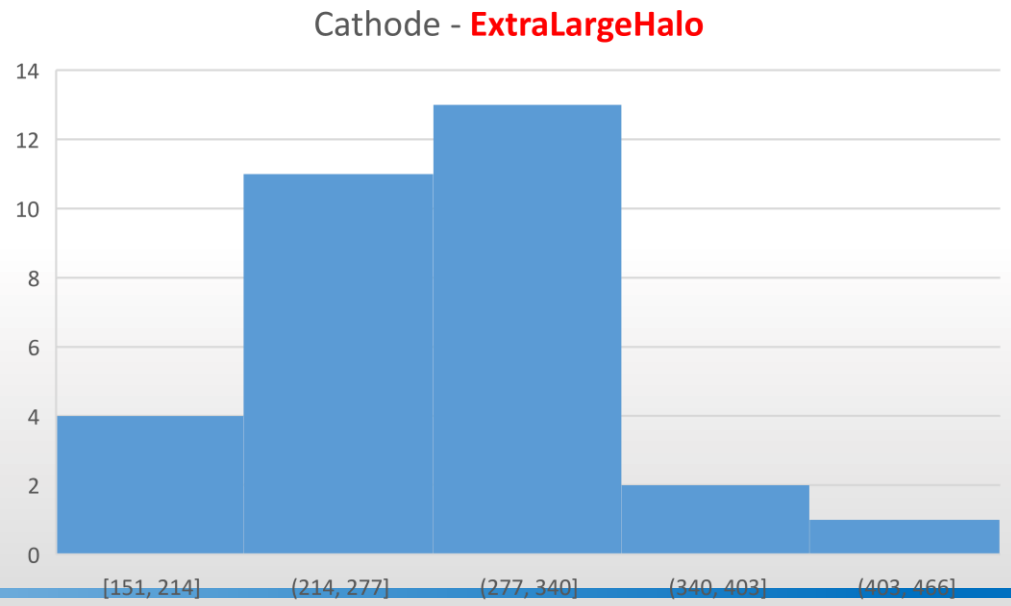
68 ÷ 293 µm

40 ÷ 173 µm

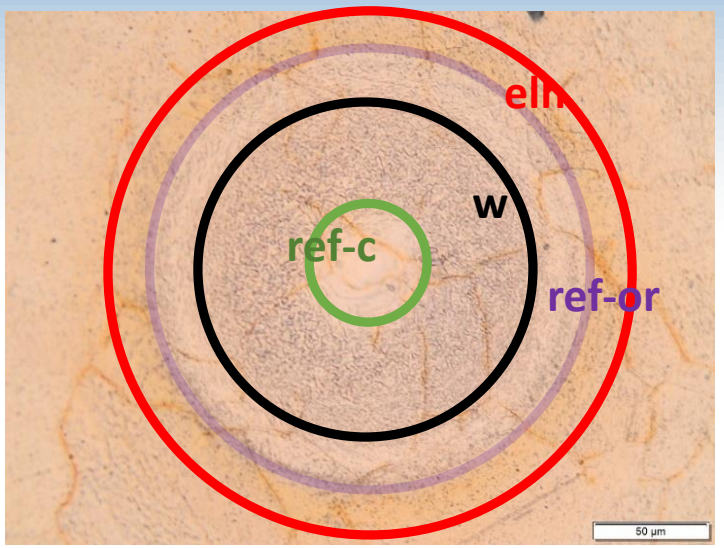
151 ÷ 425 µm

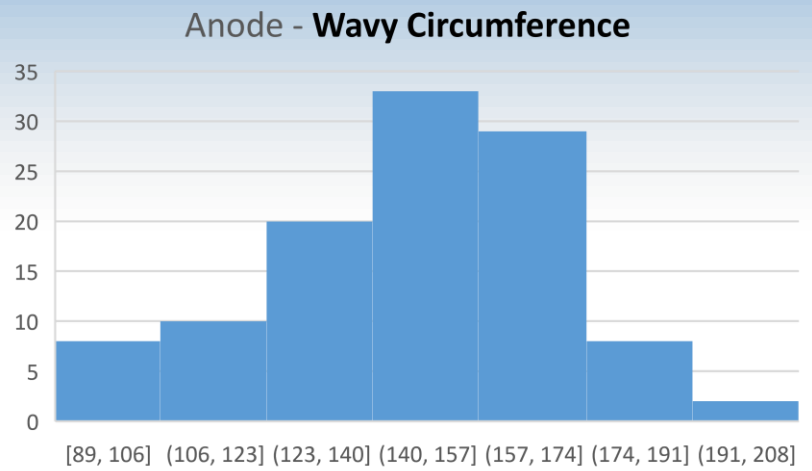


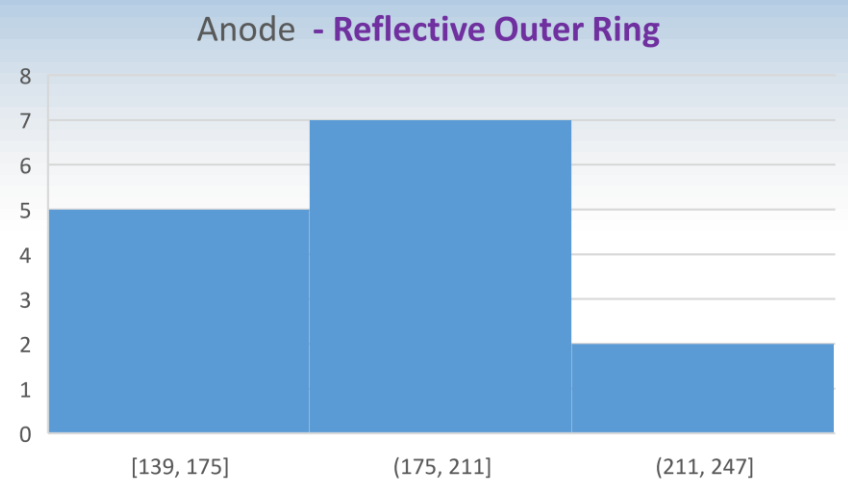


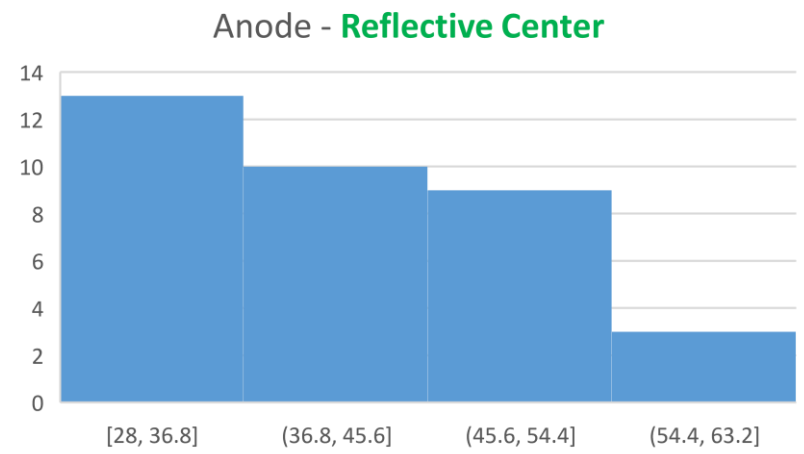


Hard Cu anode 0186-07 tested at 300 K: morphology@sizing of a typical BD

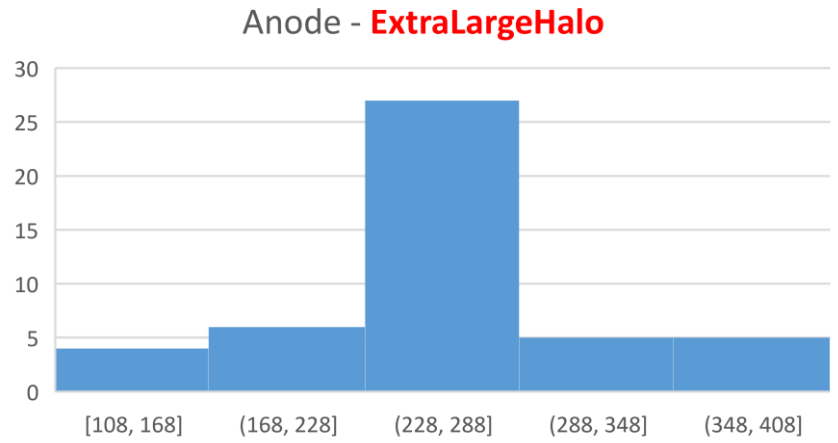




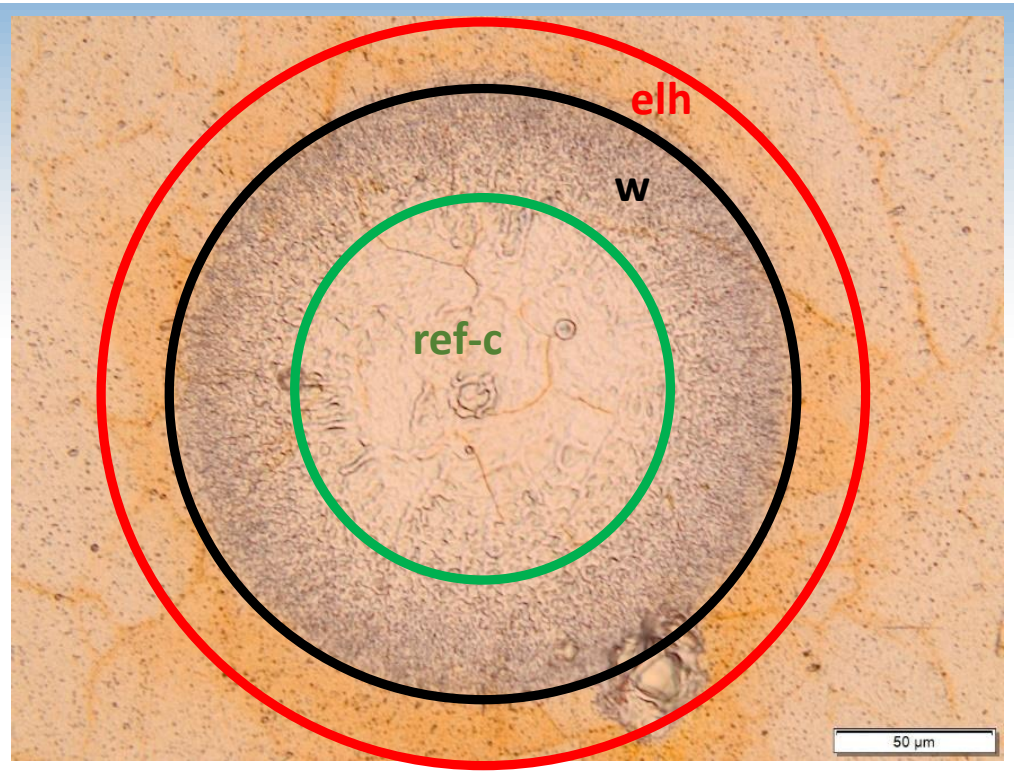




89÷ 195 µm
108÷ 396 µm
28÷ 57 µm
139÷ 219 µm



Hard Cu tested at 300 K: cathode vs anode BDs

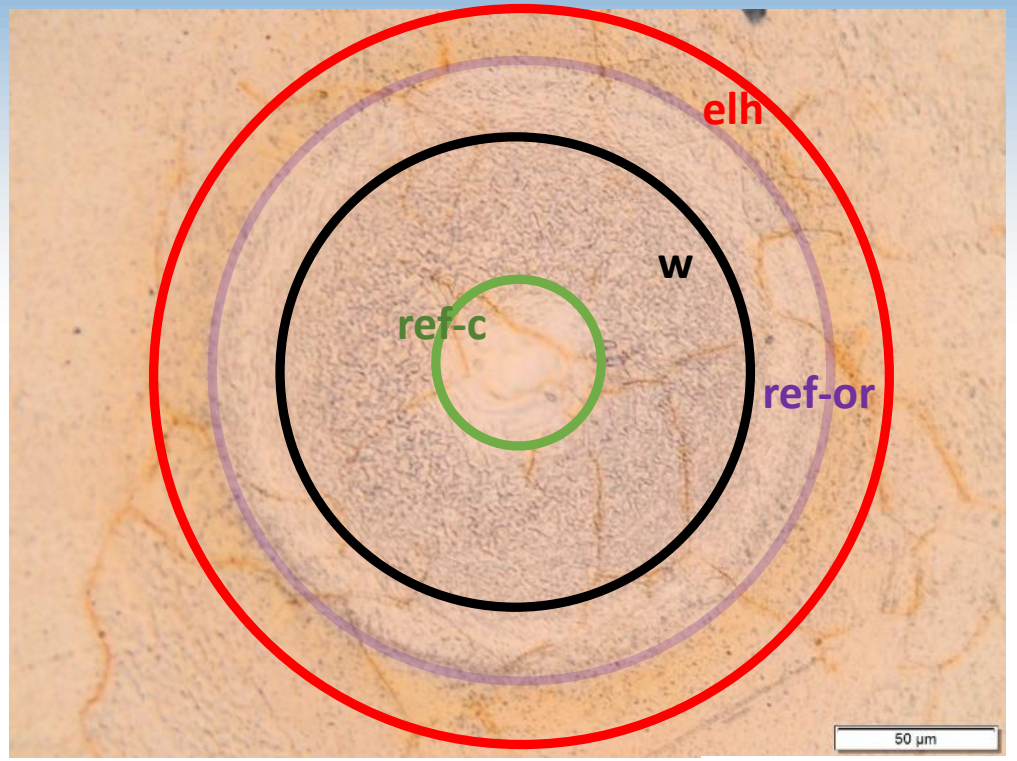


Cathode 0186-06

40 ÷ 173 µm

68 ÷ 293 µm

151 ÷ 425 µm



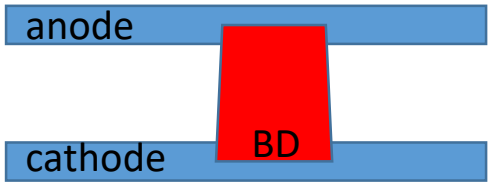
Anode 0186-07

28÷ 57 µm

89÷ 195 µm

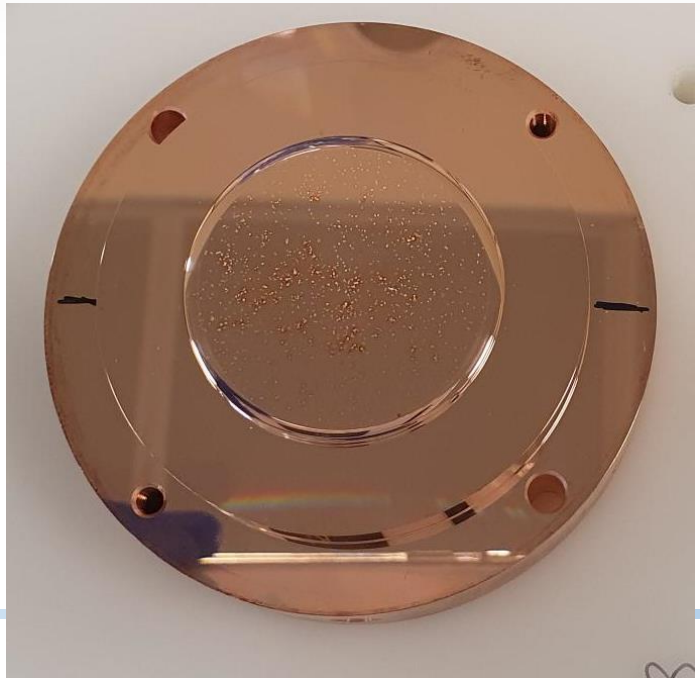
139÷ 219 µm

108÷ 396 µm

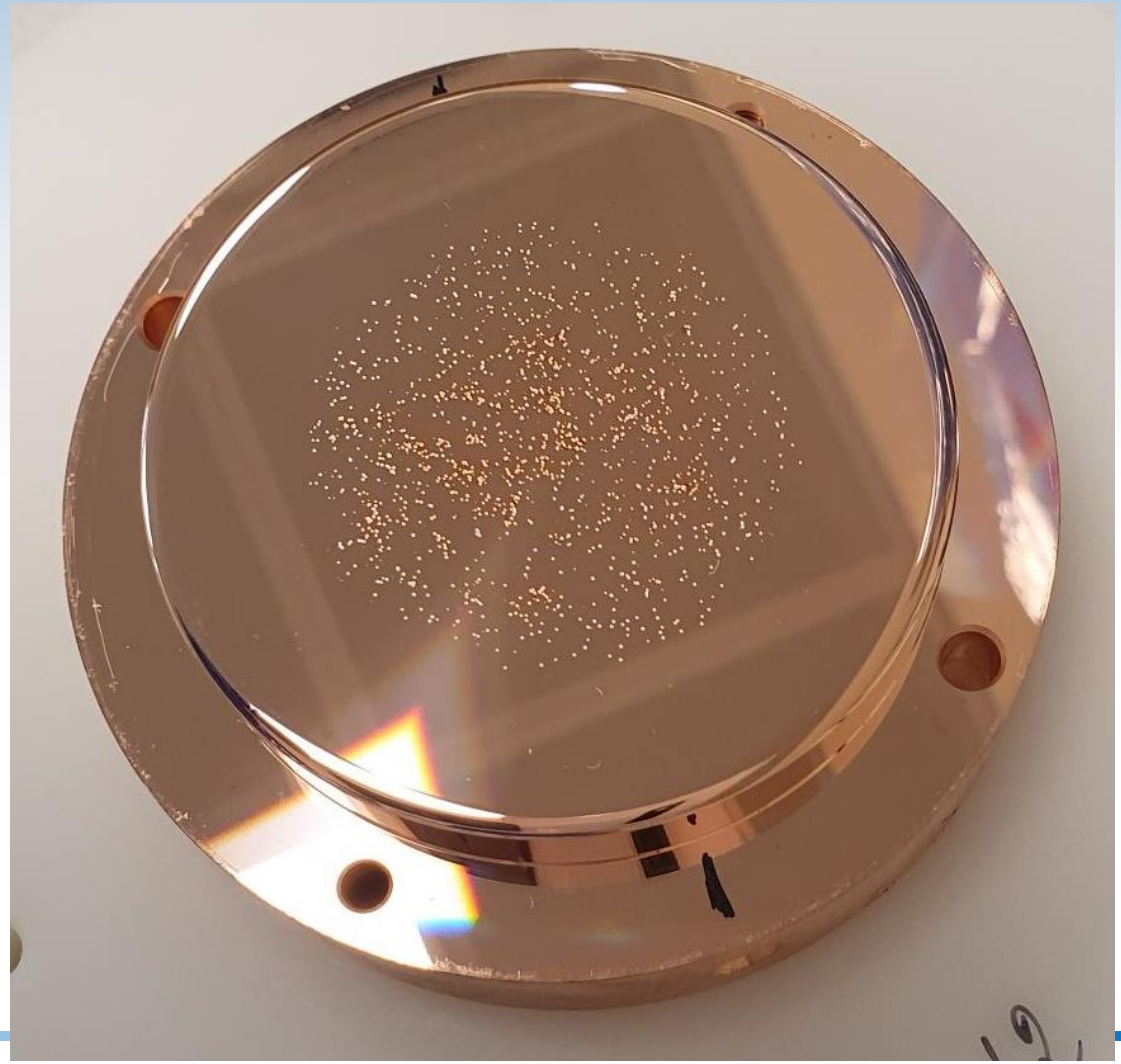


Hard Cu Electrode Pair 038 tested at 30 K: BD localization

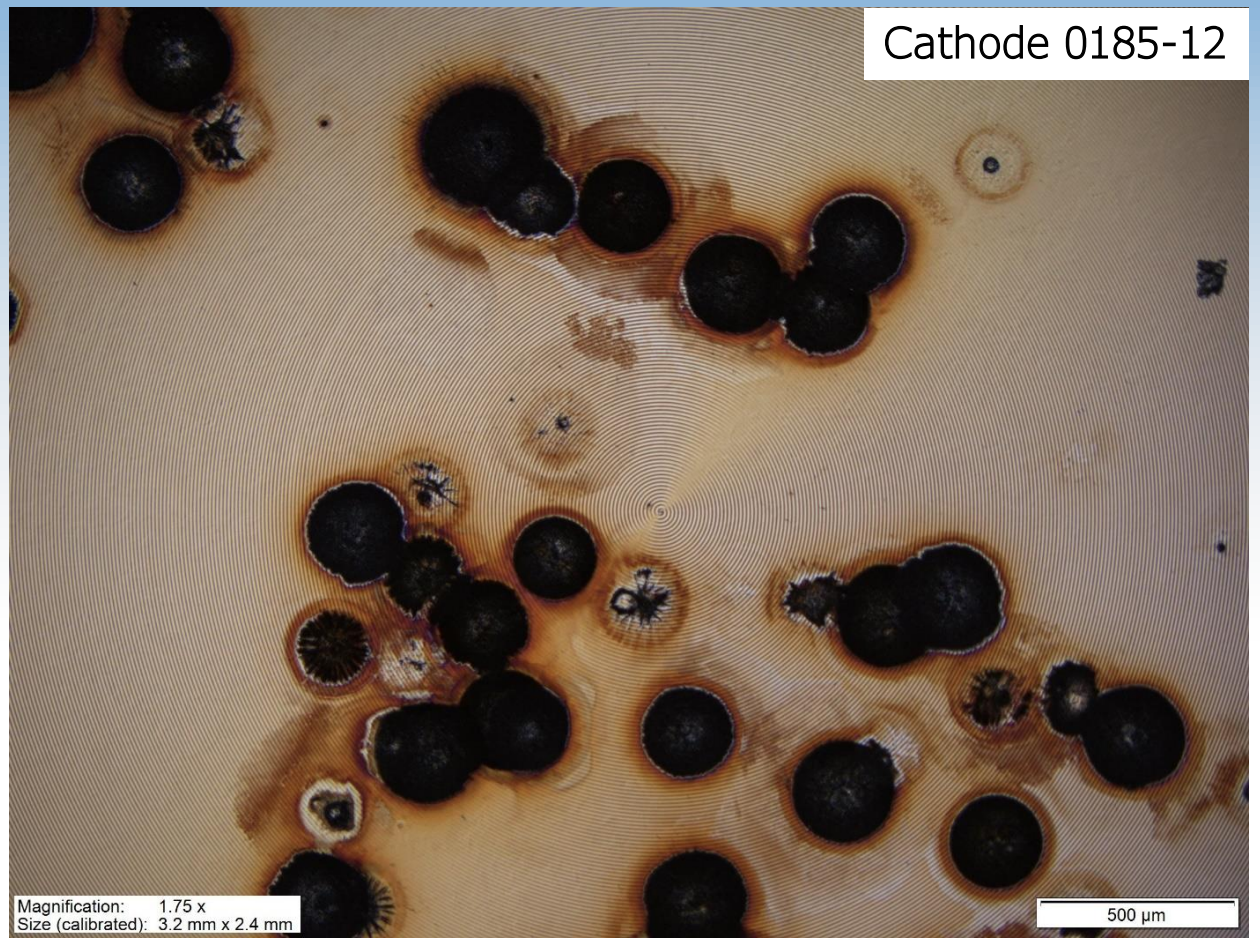
Anode 0186-13

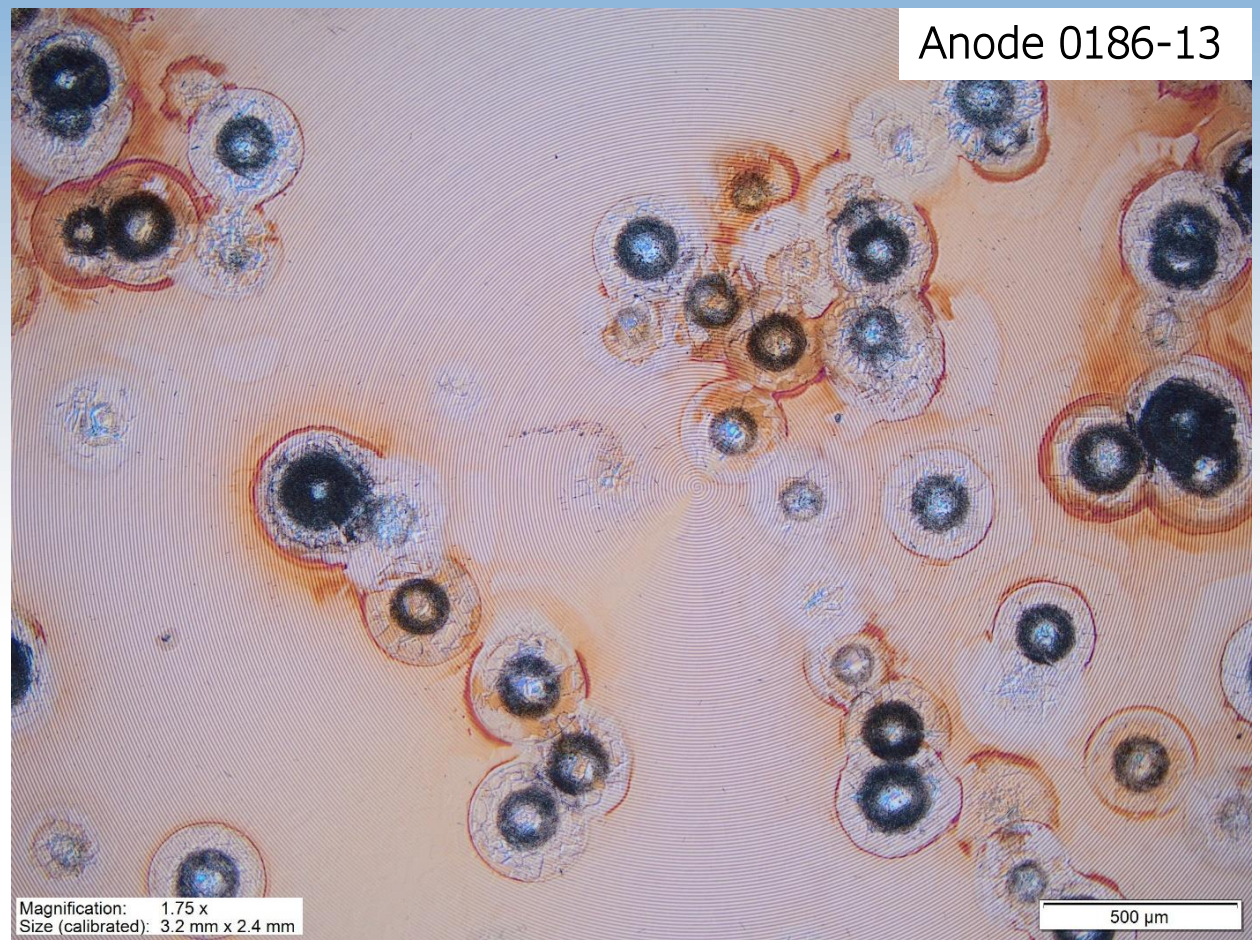


Cathode 0185-12



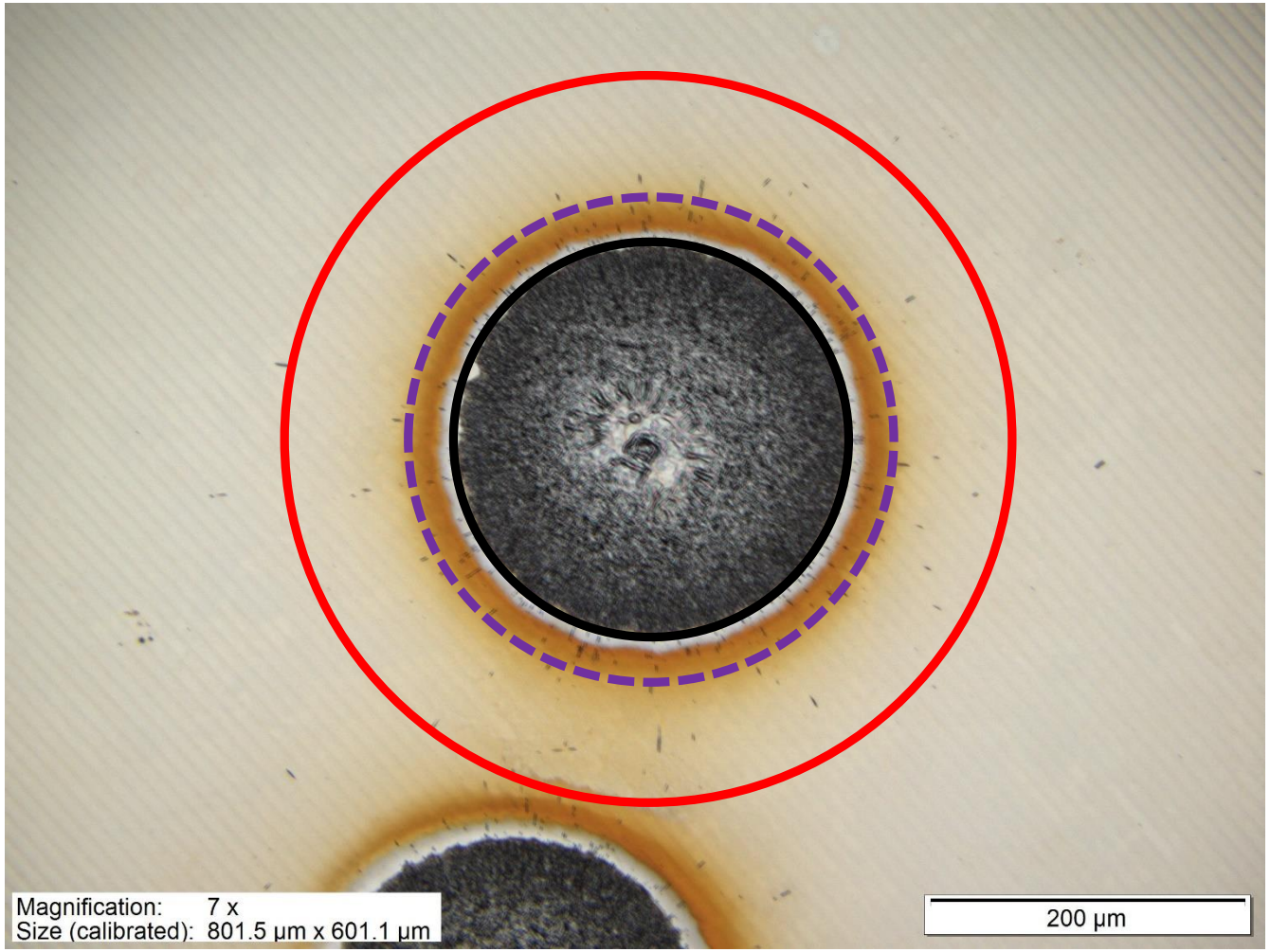
Hard Cu Electrode Pair 038 tested at 30 K: surface in LM

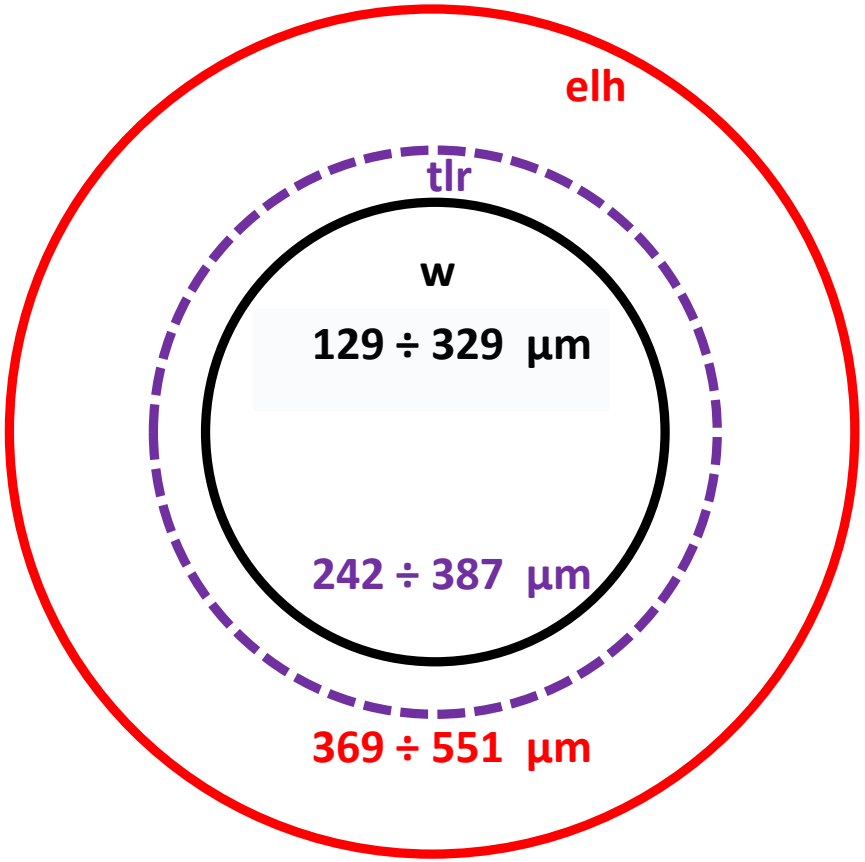




1. The grains are in the range of 20-80 µm out of the central part, 100-400 µm in the center
2. GBs are invisible except for the halo surrounding anode BDs.
3. BDs are more or less evenly distributed over the tested surfaces
4. Star-like BDs comprise at least 20% of the whole BD population

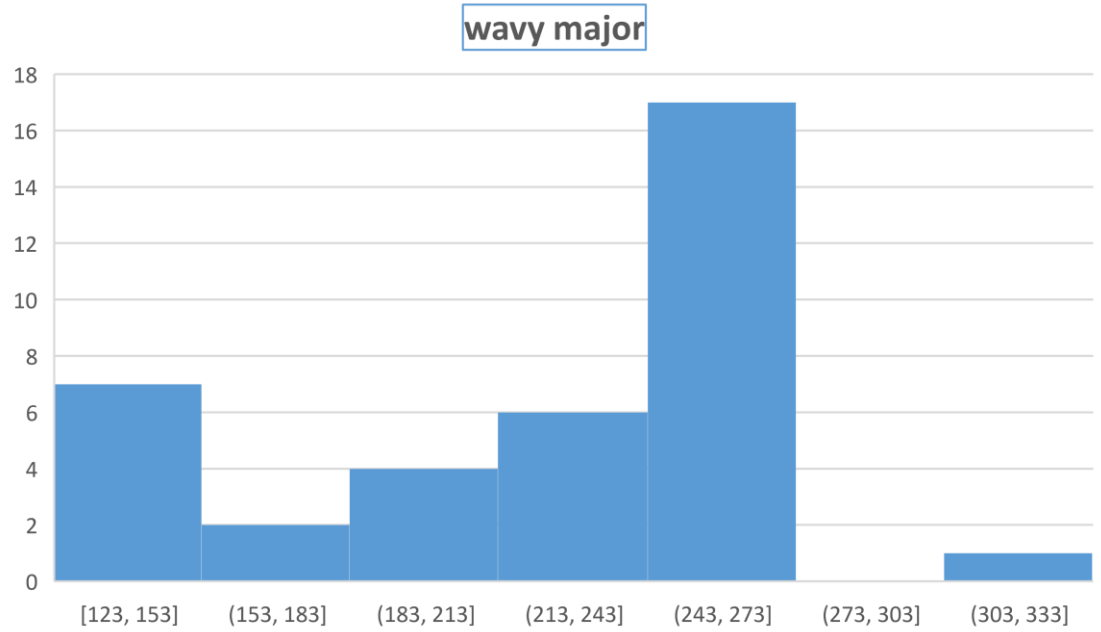
Hard Cu cathode 0185-12 tested at 30 K: morphology@sizing of a typical BD

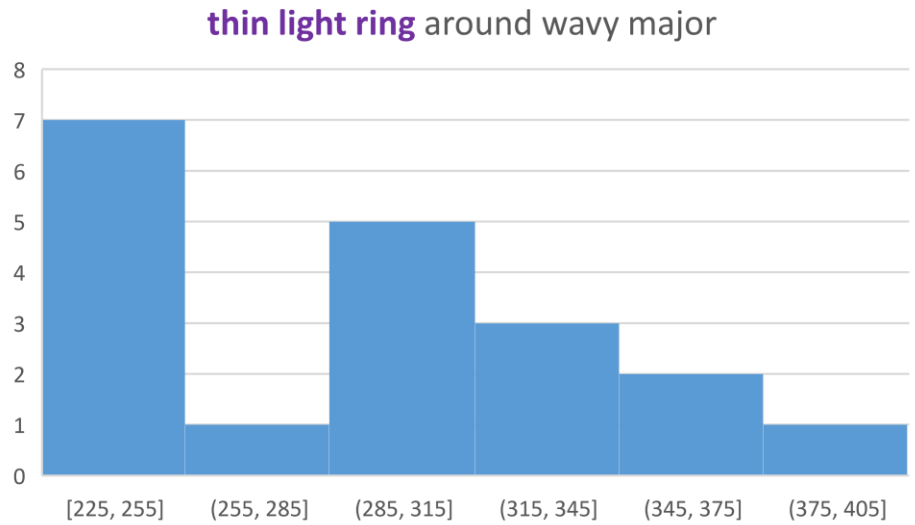


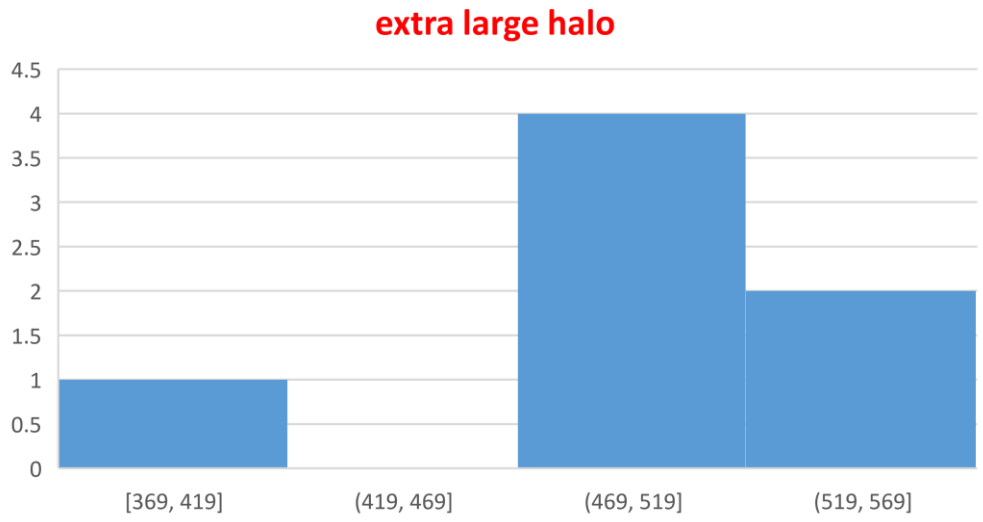


A typical round-shaped BD is composed of **a wavy dark major part (w)** surrounded by a **thin lighter ring** which may be surrounded by a ring of **an extra-large halo**

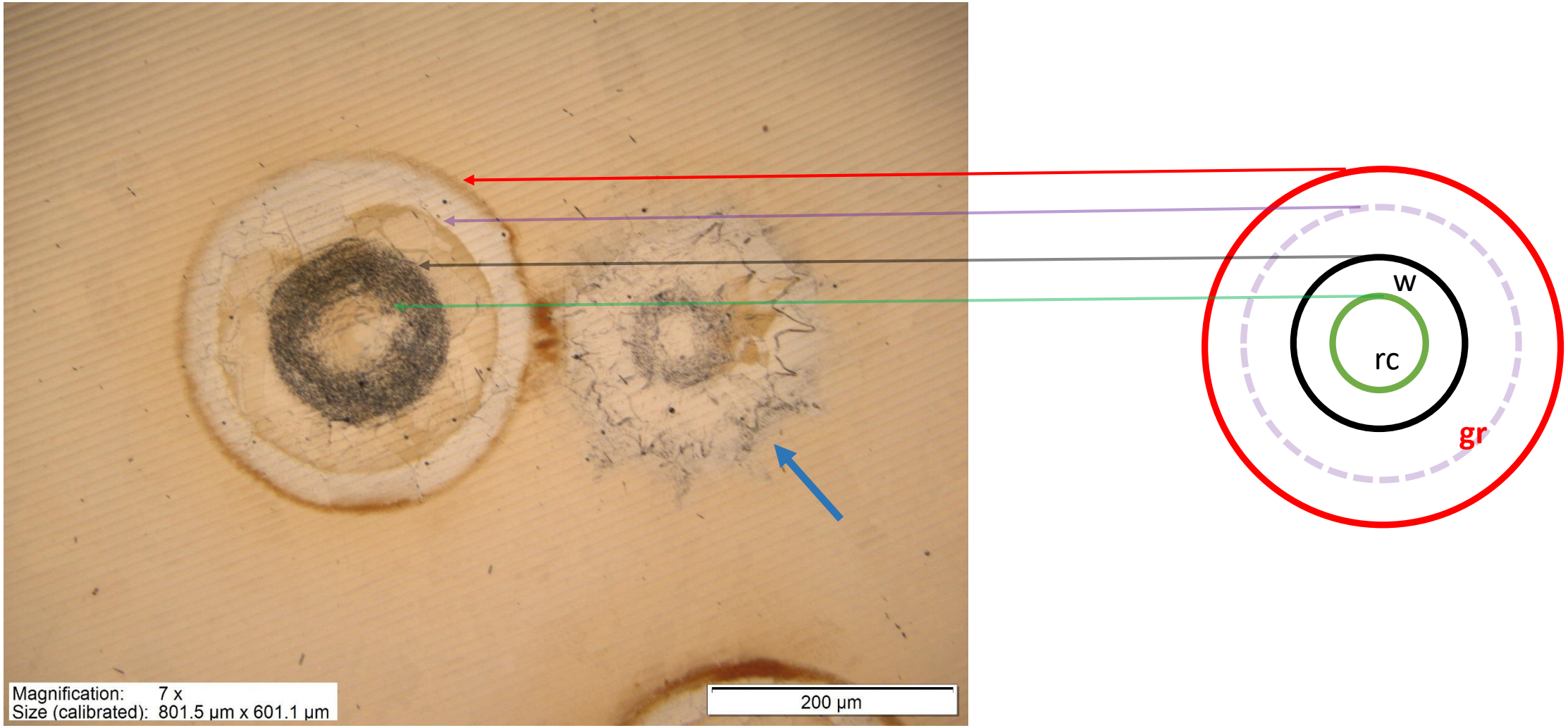
Hard Cu cathode 0185-12 tested at 30 K: morphology@sizing of a typical BD





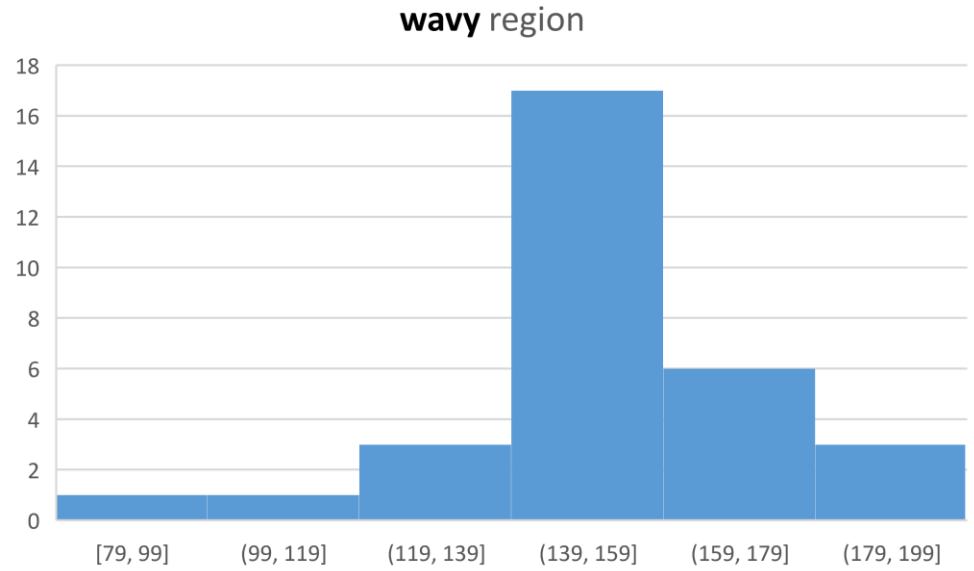


Hard Cu anode 0186-13 tested at 30 K: morphology@sizing of a typical BD



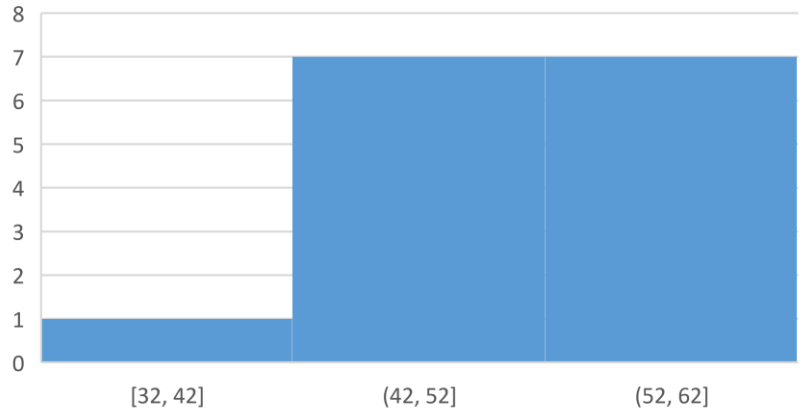
A typical round-shaped anode BD is composed of **a reflective central part (rc)** surrounded by **a wavy circumference (w)** surrounded by **a wide grainy halo (grh) of etched GBs**

Hard Cu anode 0186-13 tested at 30 K: morphology@sizing of a typical BD



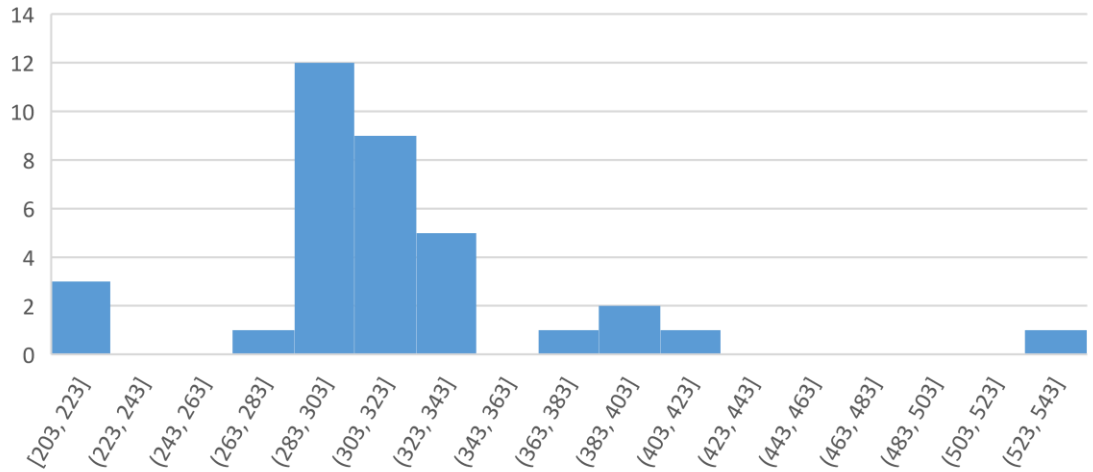
w = 79 – 188 µm

reflective center



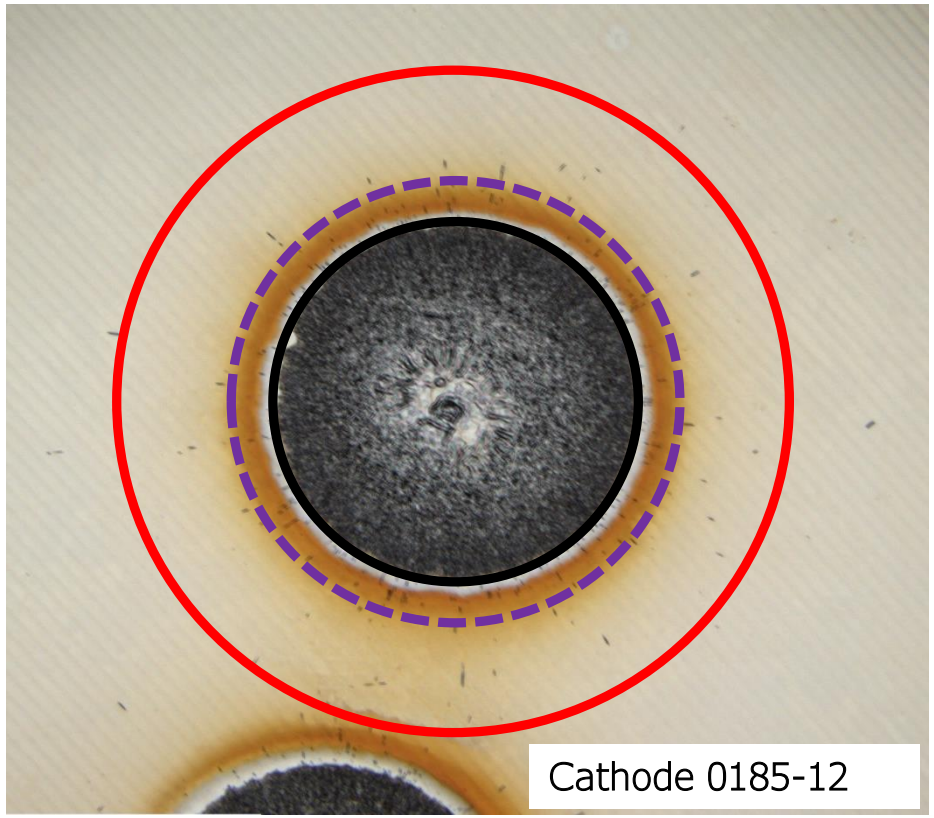
rc = 32 – 50 µm

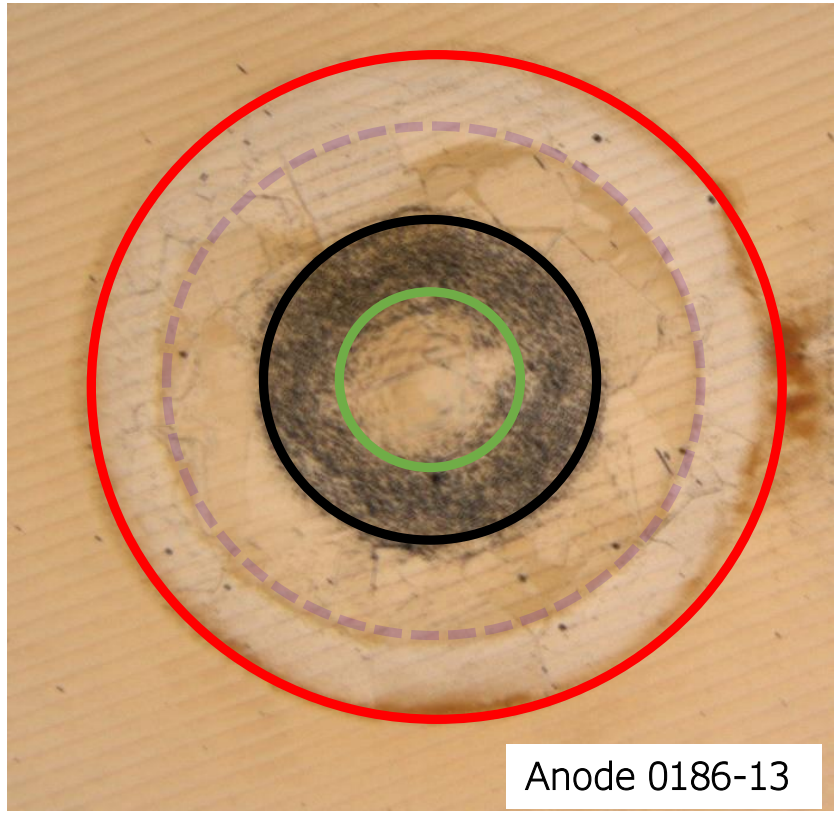
grainy halo



grh = 294 – 536 µm

Hard Cu tested at 30 K: cathode vs anode BDs



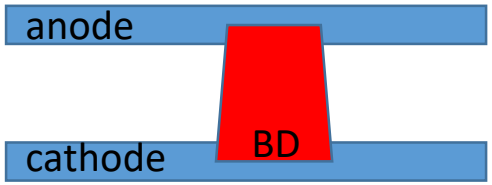


32 ÷ 50 µm

129 ÷ 329 µm

79 ÷ 188 µm

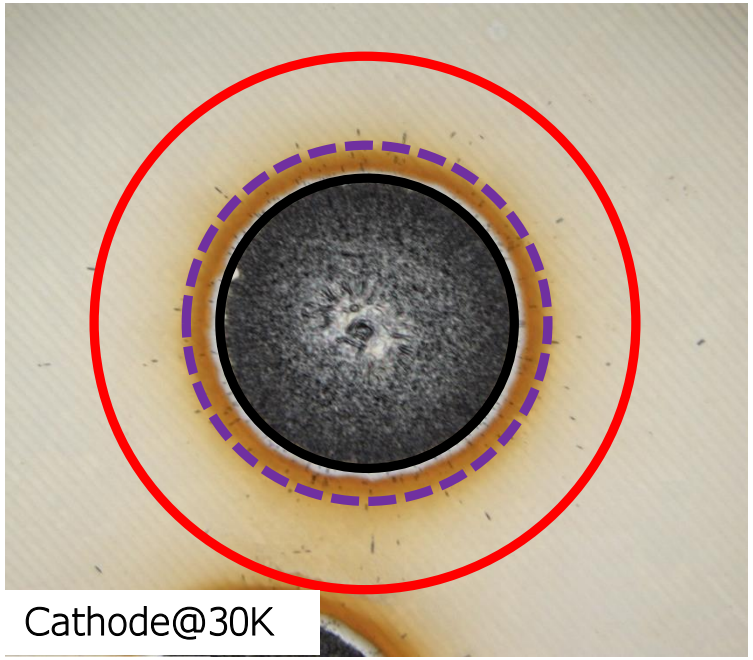
242 ÷ 387 µm



361 ÷ 551 µm

294 ÷ 536 µm

Hard Cu : cathode@30 K vs cathode@300 K



no reflective center

129 ÷ 329 µm

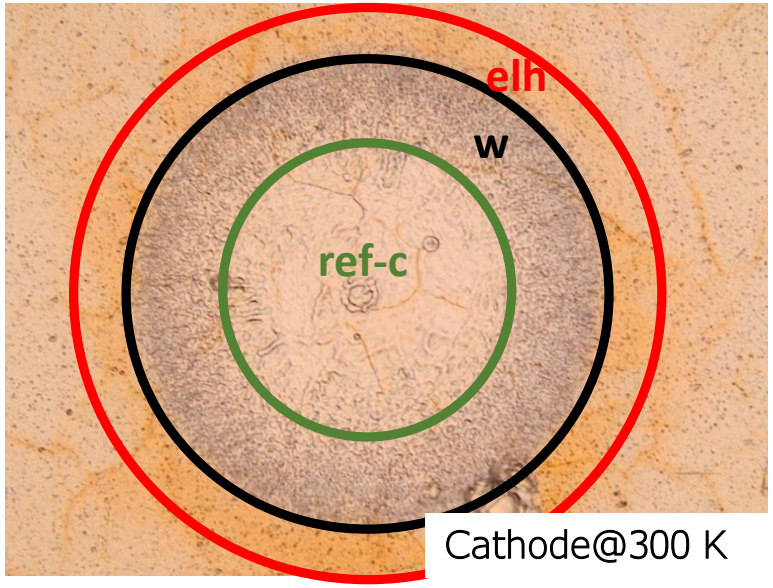
242 ÷ 387 µm

361 ÷ 551 µm

40 ÷ 173 µm

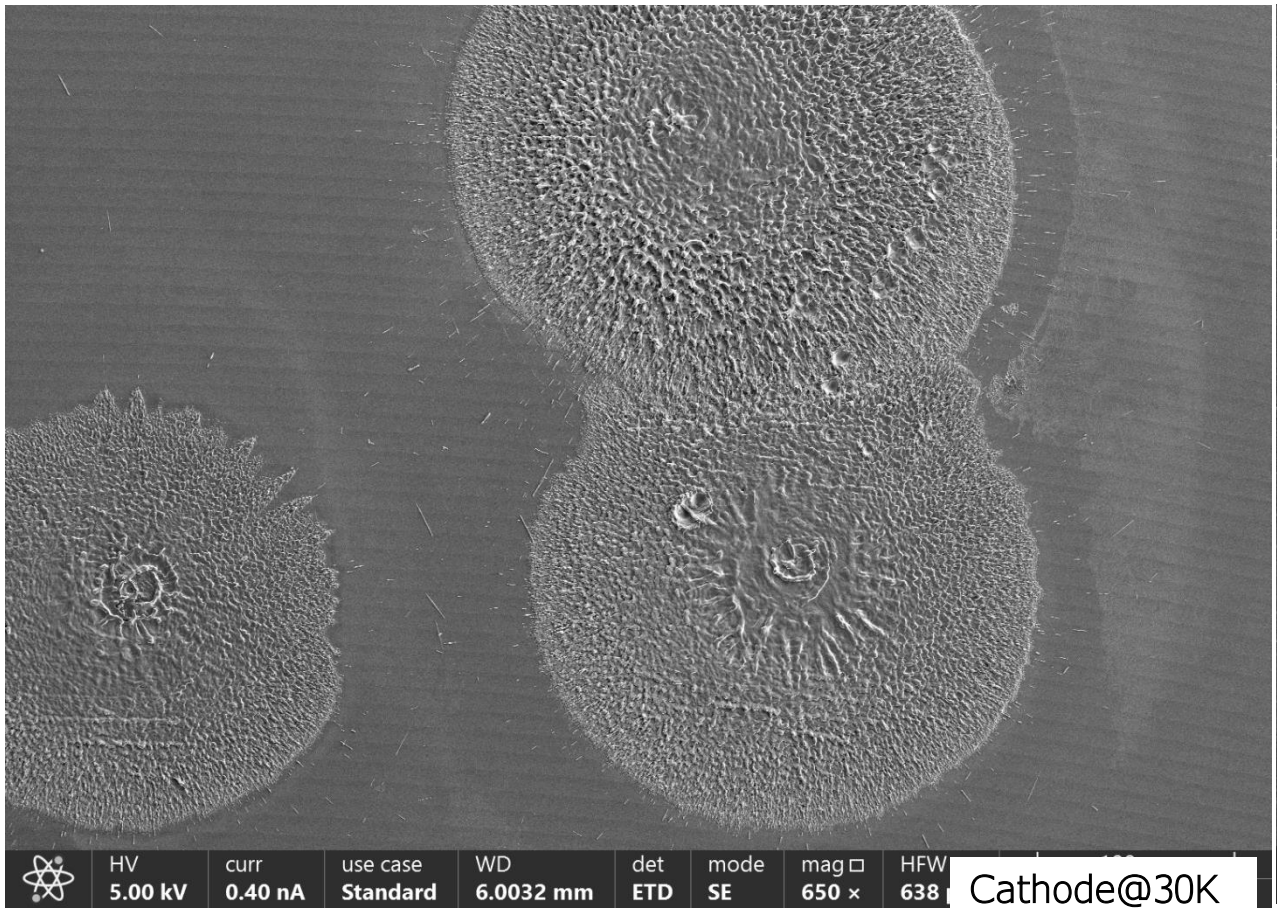
68 ÷ 293 µm

151 ÷ 425 µm

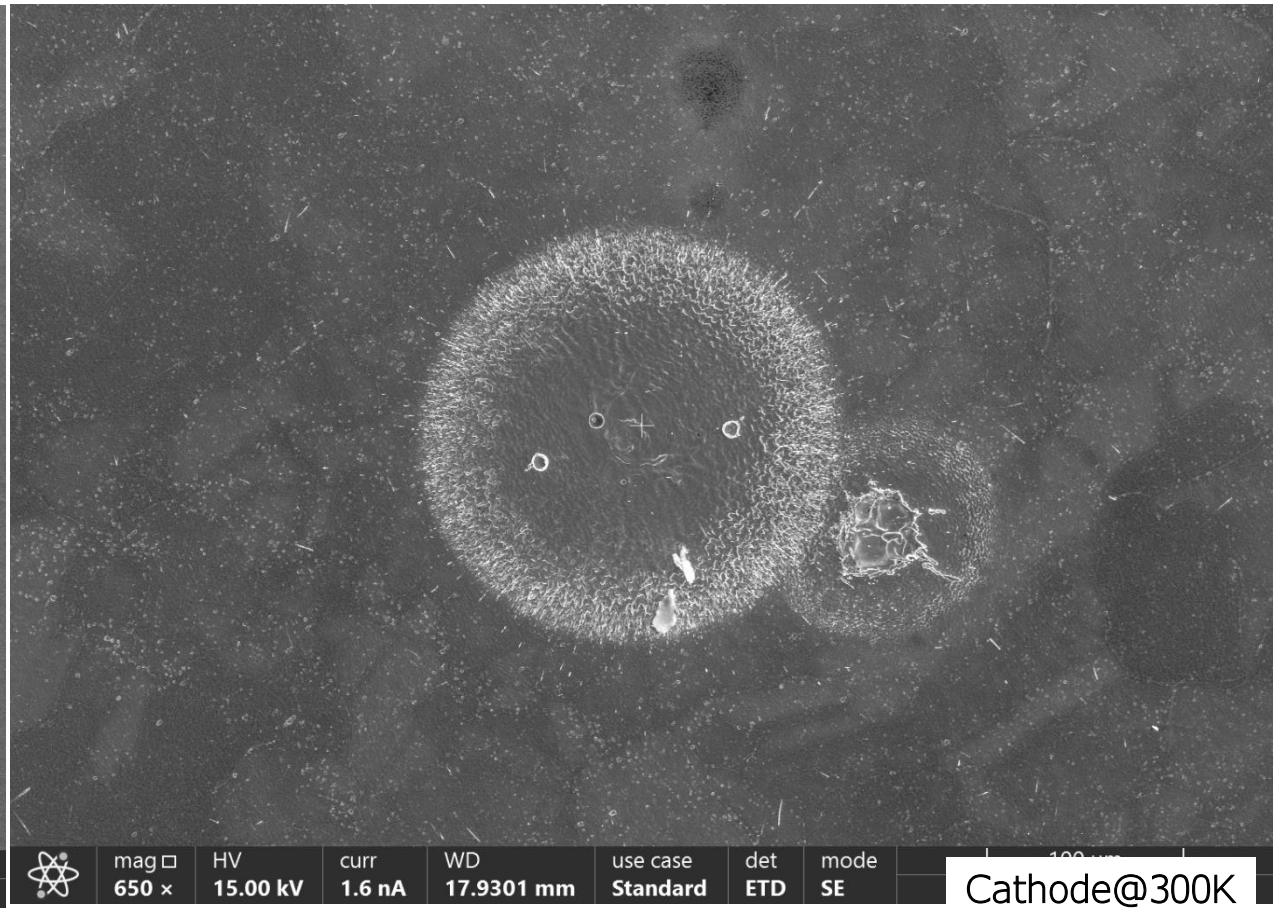


- A typical round-shaped cathode BD@30K is larger and more rough than BD@300K

Hard Cu : cathode@30 K vs cathode@300 K - differing topography



Cathode@30K

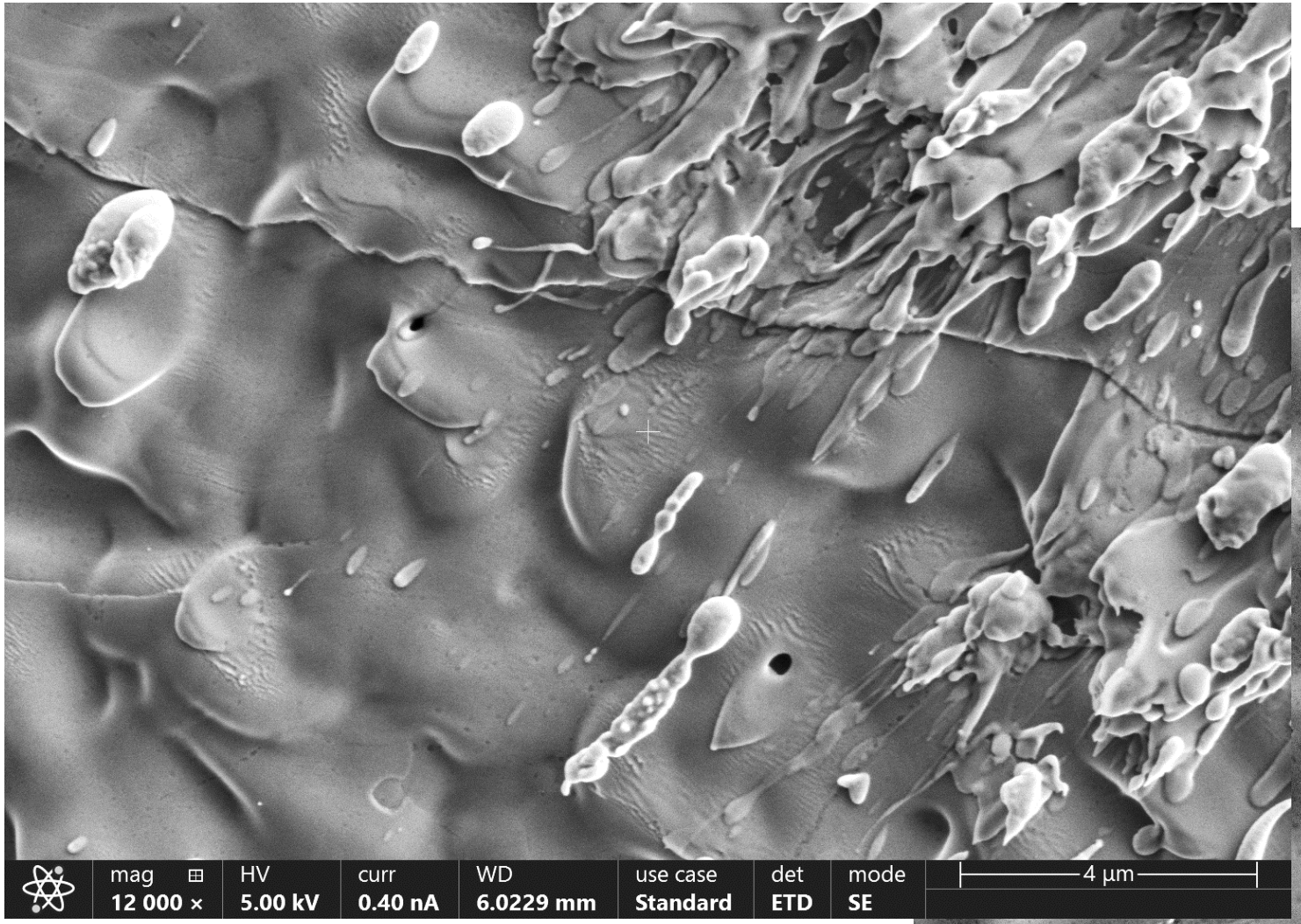


Cathode@300K

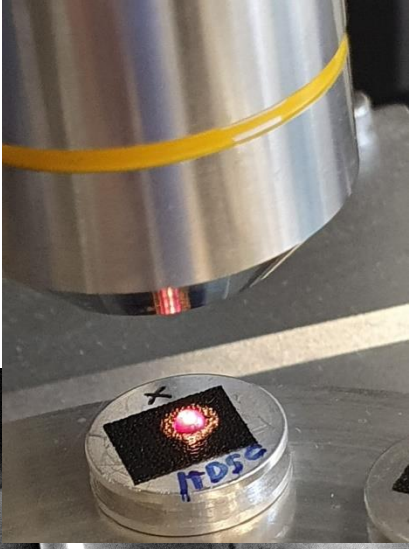
- A typical round-shaped cathode BD@30K is:
  - larger than BD@300K
  - more rough that BD@300K
  - doesn't have a reflective center

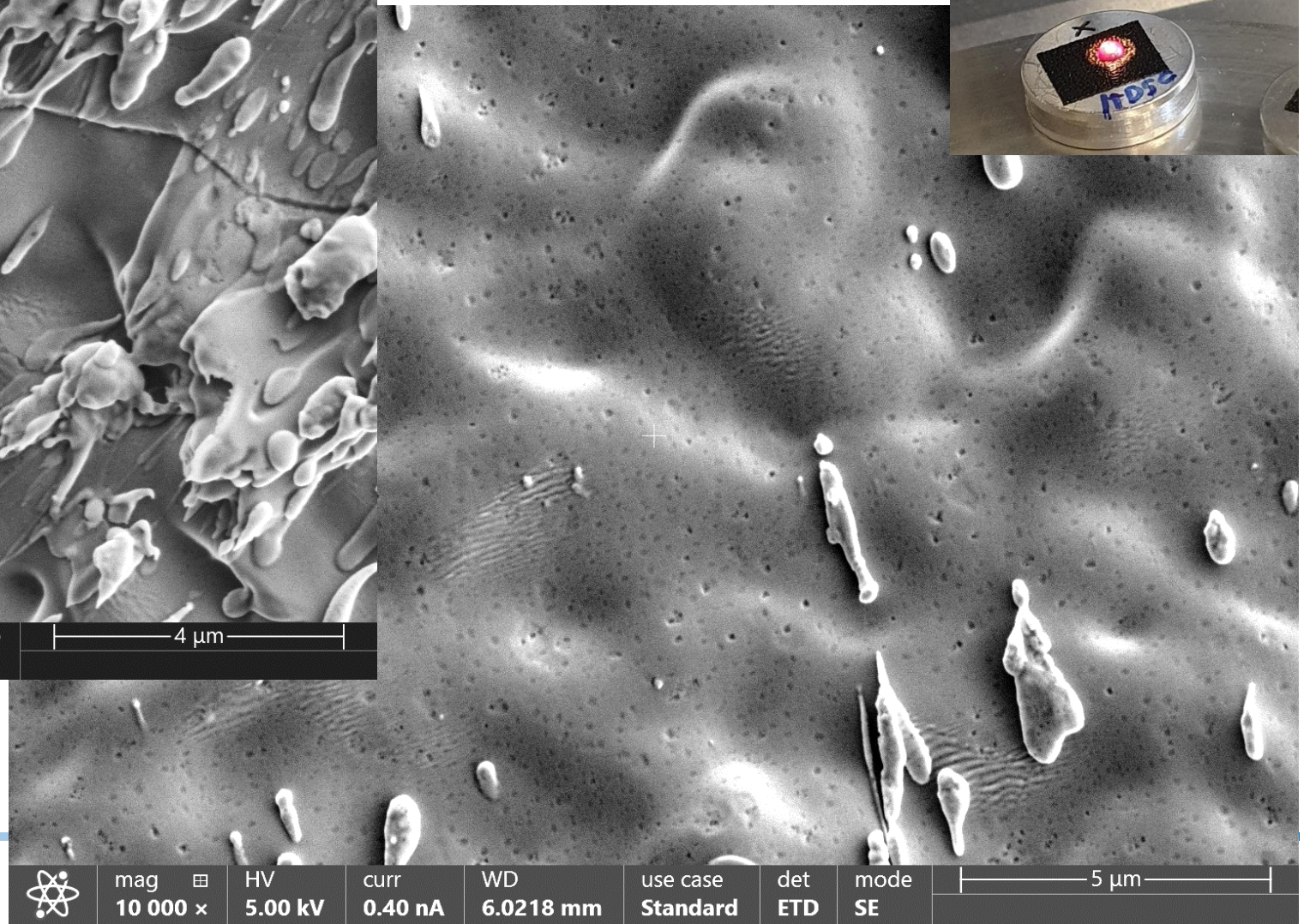
- ✓ Cathode BD@300K is more relaxed
- ✓ Cathode BD@30K are frozen/thermally shocked

Hard Cu : cathode@300 K - SPR – resonance –related features

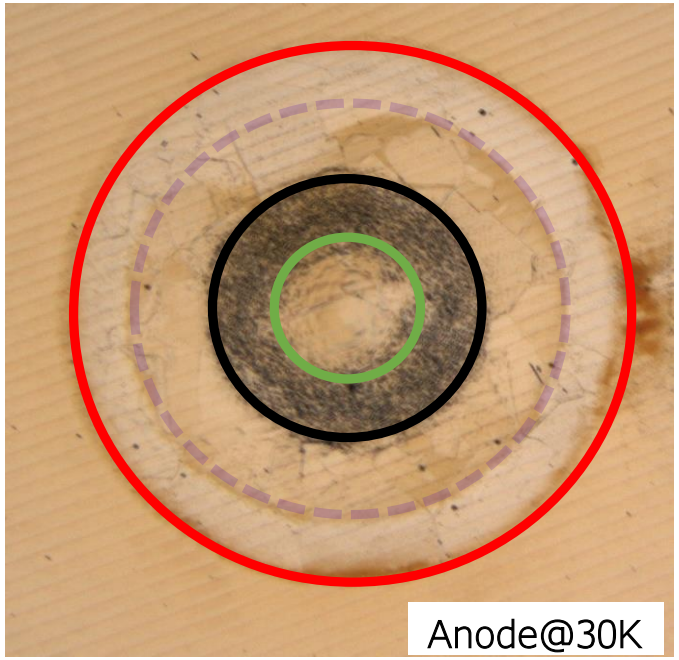


70 ÷ 130 nm spacing





Hard Cu : anode@30 K vs anode@300 K



32 ÷ 50 µm

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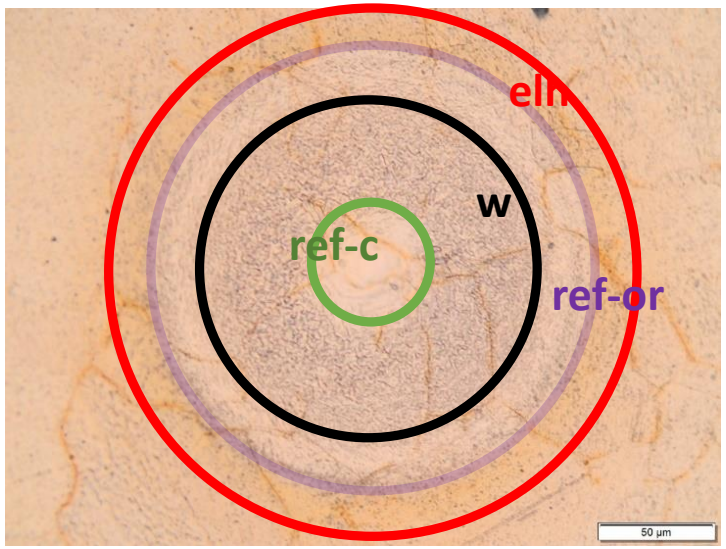
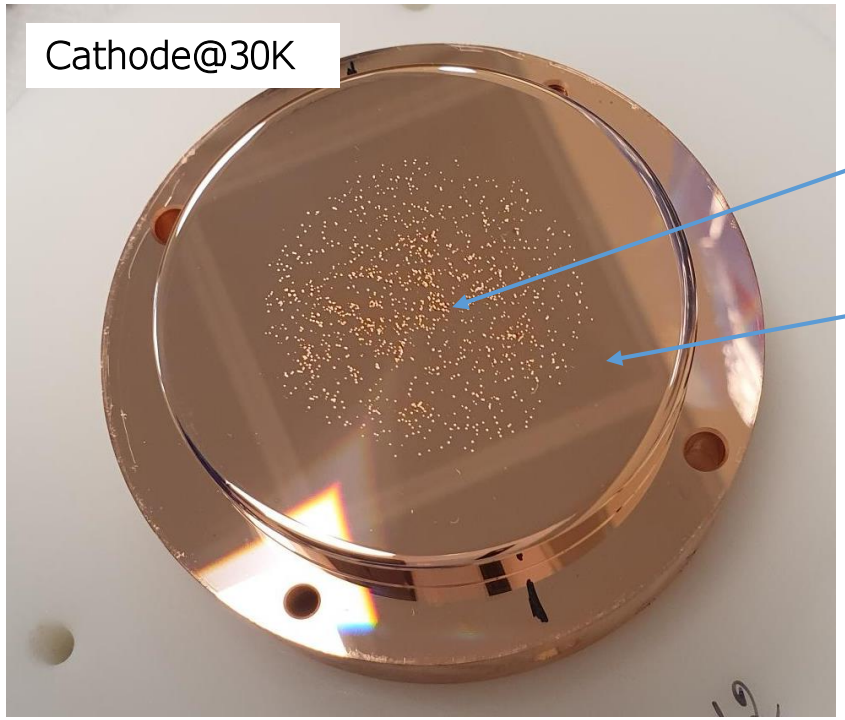


Anode@300K

- Typical round-shaped anode BDs are of almost the same size for 30 and 300K
- GB etching is observed only at anode@30K (anode@300K was pre-etched)

Hard Cu cathode@30 K and cathode@300 K - a subsurface structure via FIB/TEM



Field – Exposed region

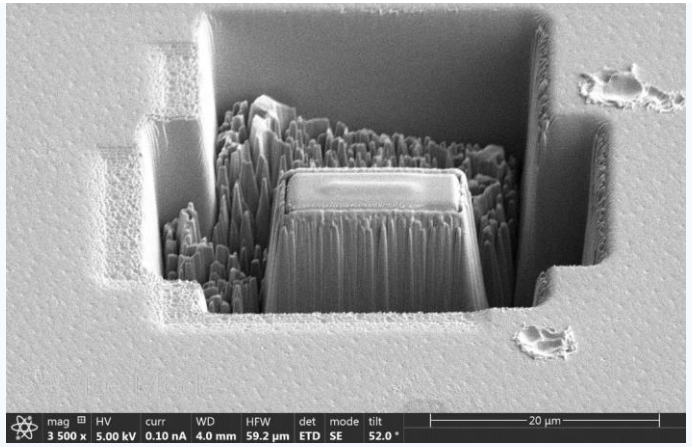
Reference region

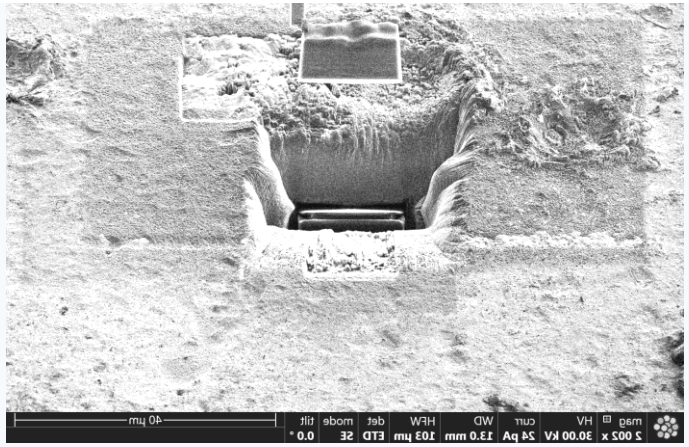
✓ We extract lamellas in blind since grain boundaries are invisible in the Ref region and hardly visible in the Field-exposed region.

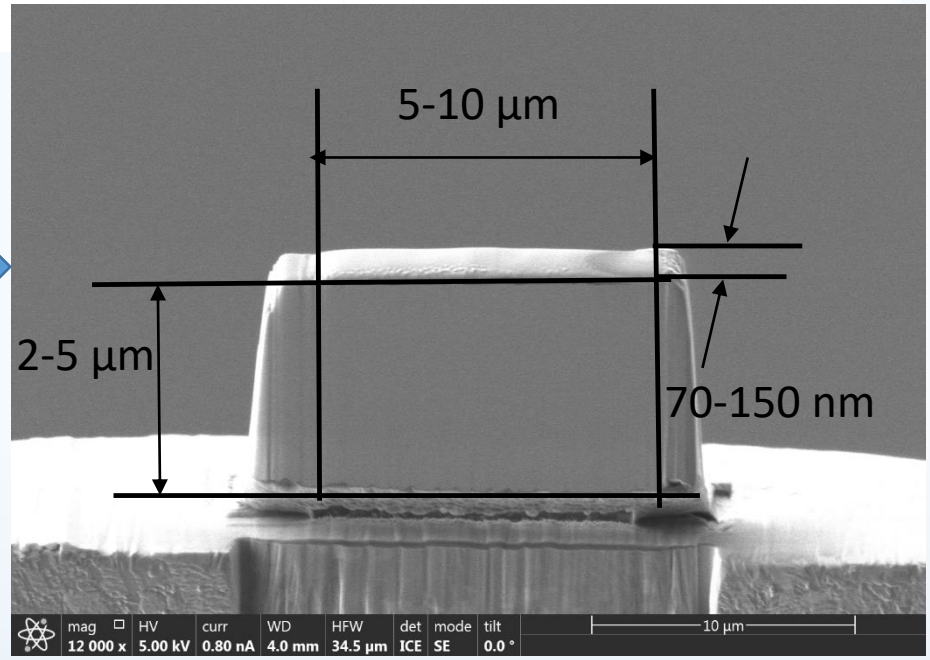
✓ We extract lamellas from a grain body trying to avoid GBs and deep etching pits.

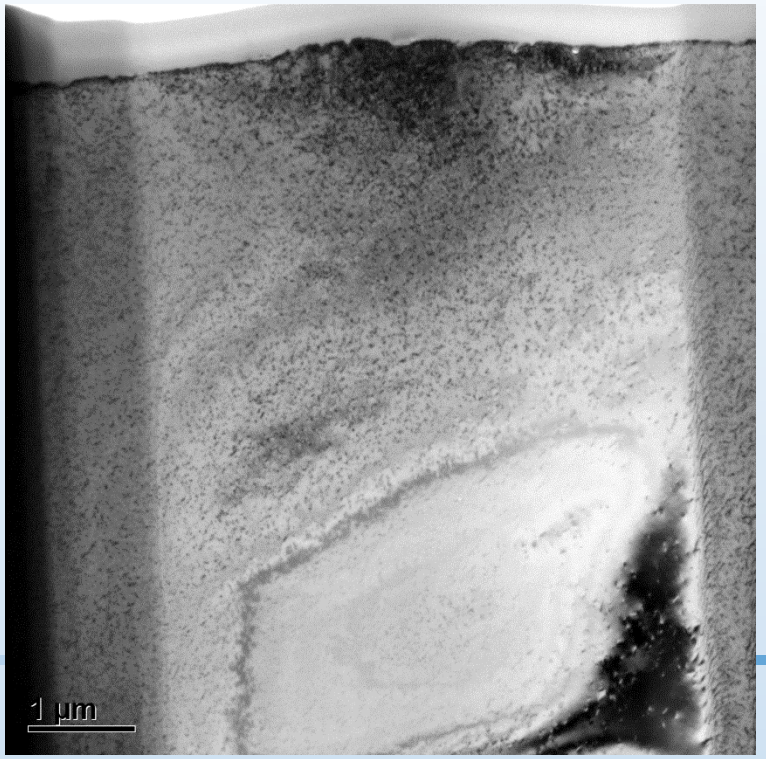
- We extracted cross-sectional samples with FIB, i.e. the ion beam was normal to the electrode surface. The sites were chosen millimeters away one another in Ref region and sub-millimeter away in FE .
- To avoid post-preparation deformation of a thin membrane we extract lamellas of maximum width (7-8 μm) and reasonable thickness 100-150 nm.
- We focus out attention of the outer ~ 500 nm subsurface region because namely this part of electrode should mainly be affected by the electrical field.

Hard Cu, cathode@30 K and cathode@300 K - a subsurface structure via FIB/TEM





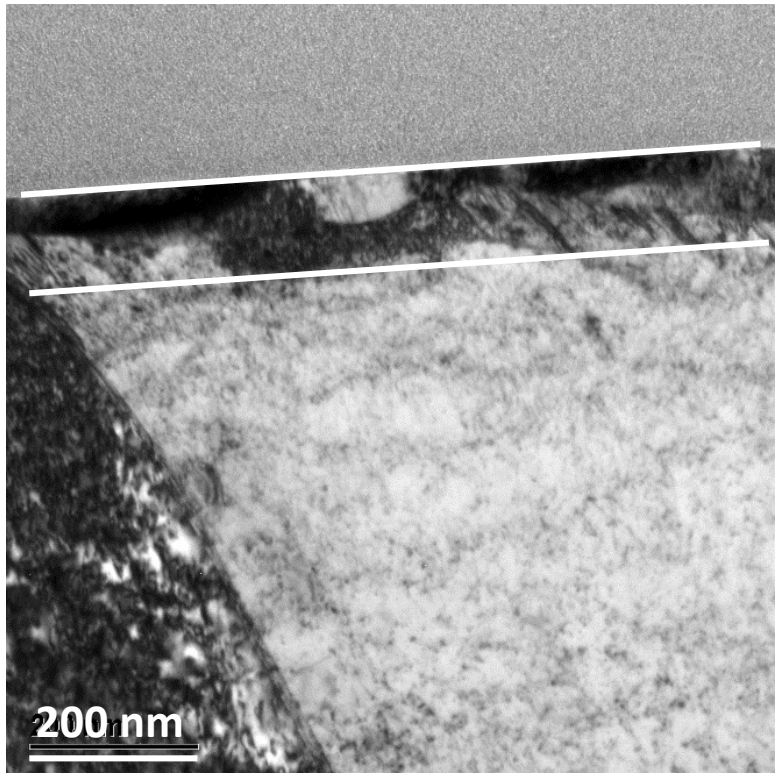


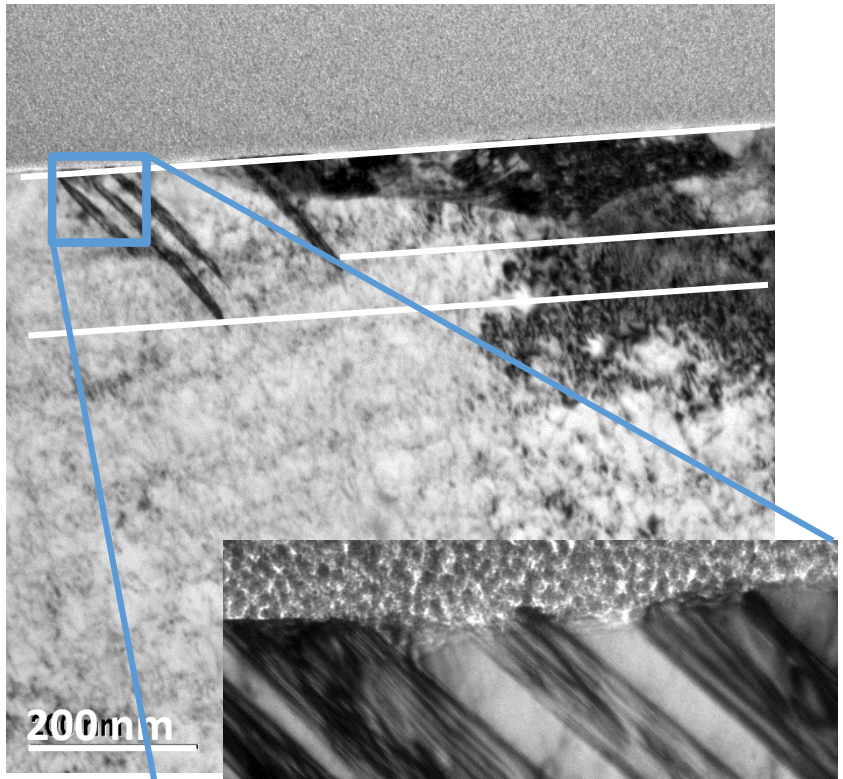


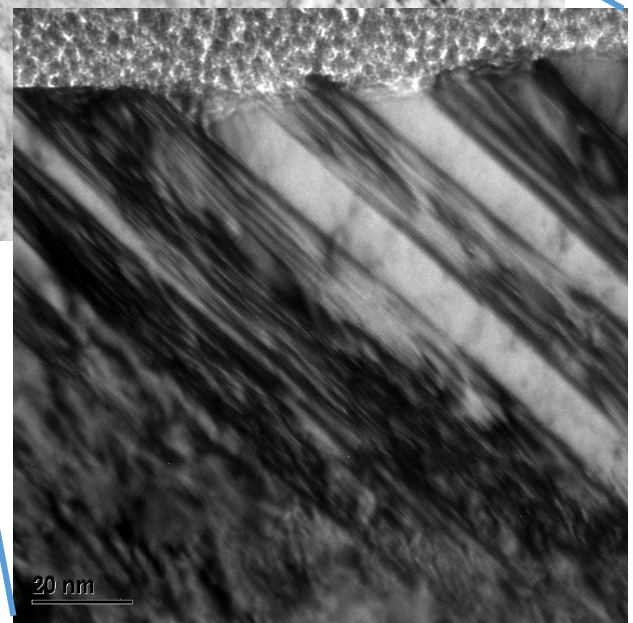
BF TEM image of a FIB-thinned transparent window (TEM lamella)

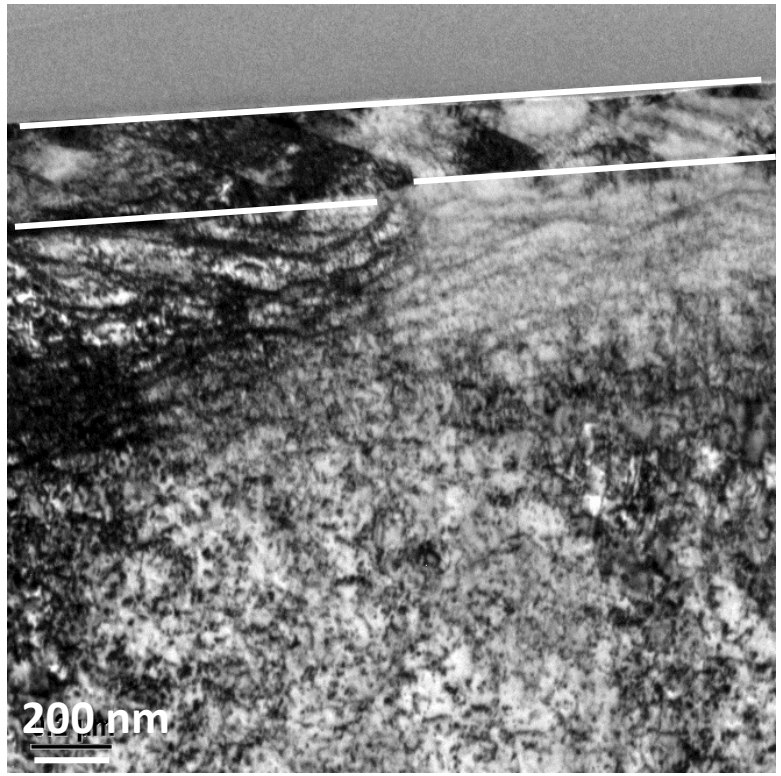
upmost ~ 500nm

A surface hardening is visible down to < 300 nm (sometimes < 200 nm only)



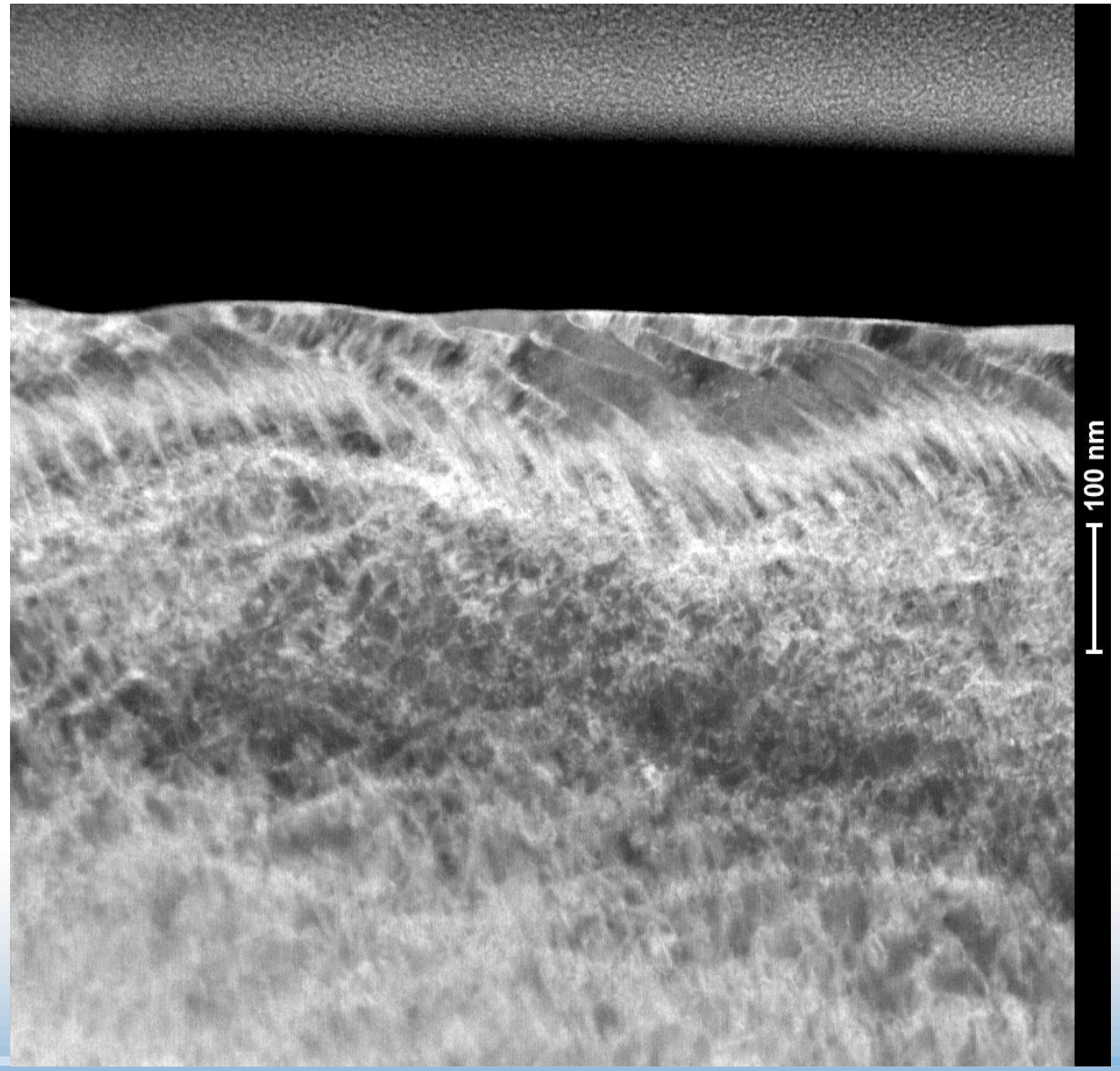


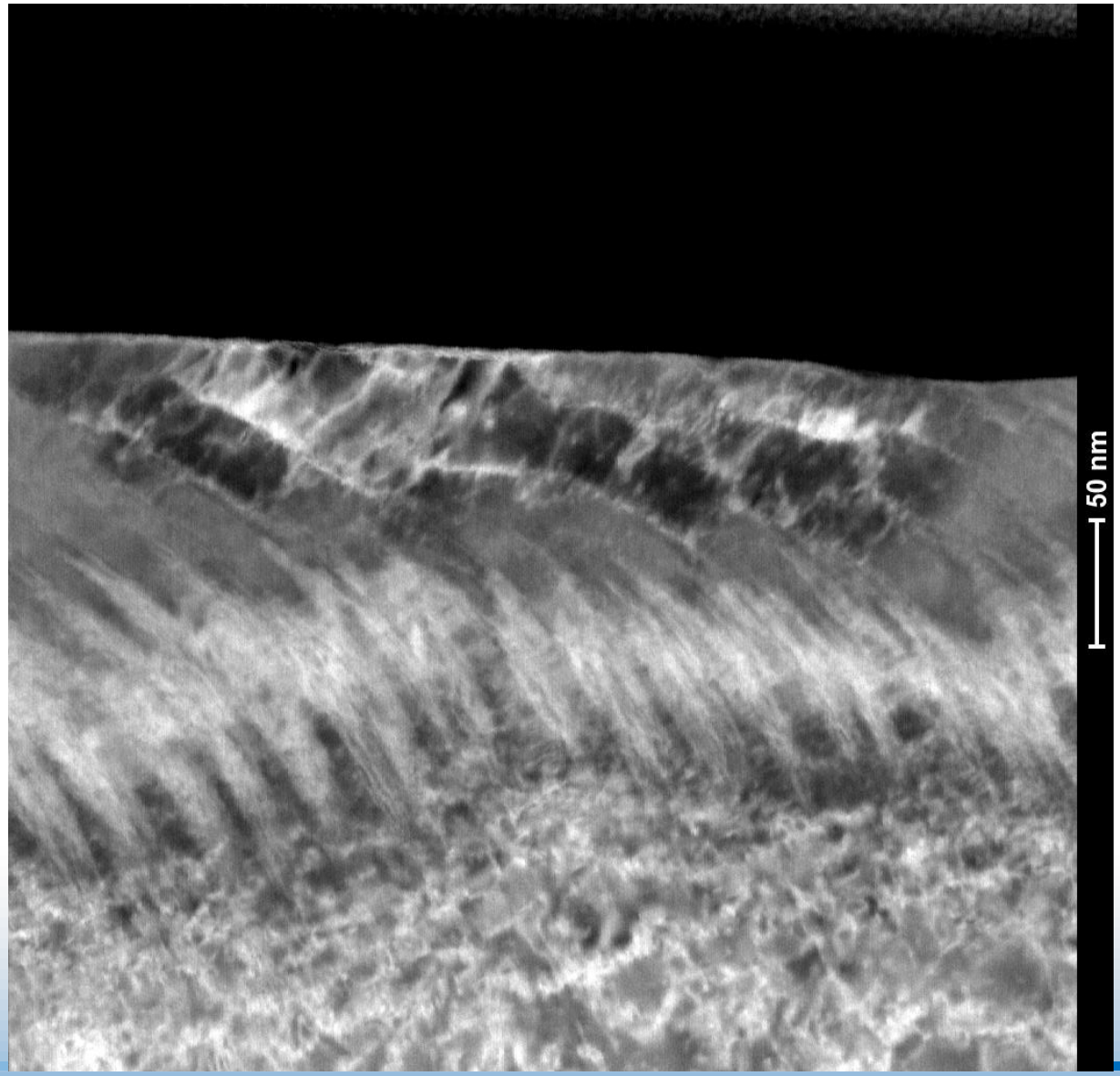




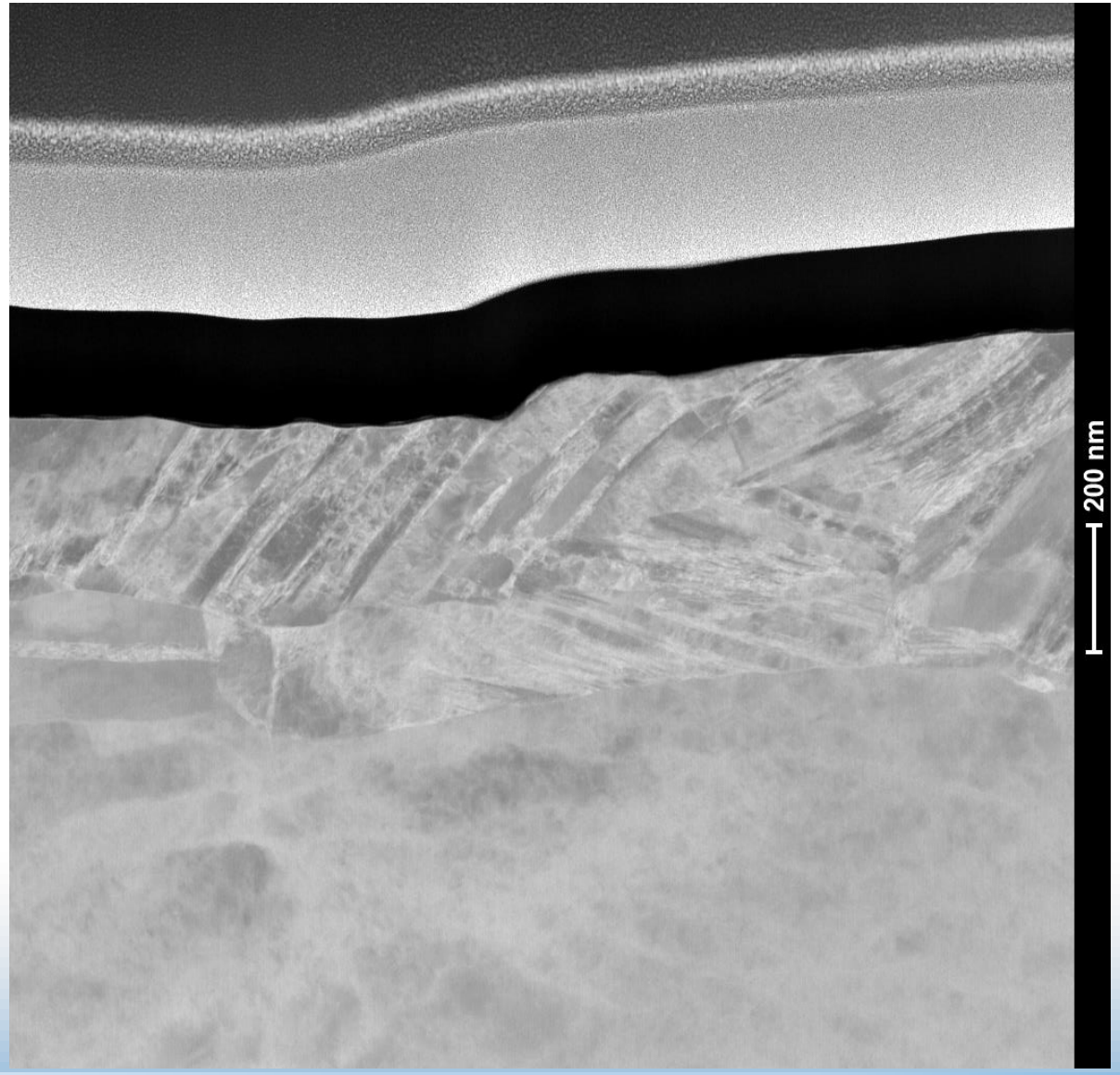
- very thin twins (~ 10- 20 nm)

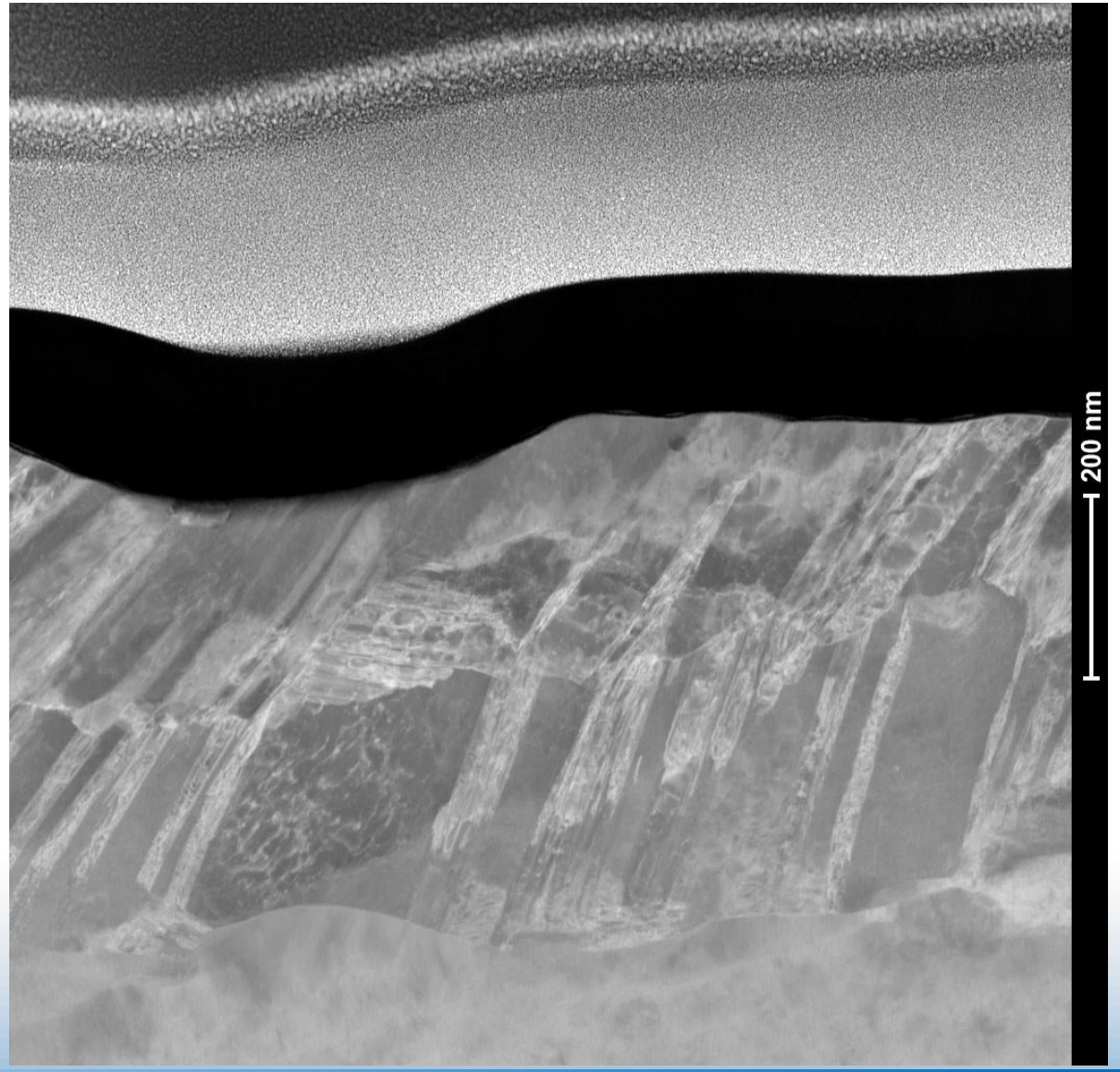
Hard Cu cathode@30 K – Reference Region: extended twinned structures within the upmost ~ 500nm



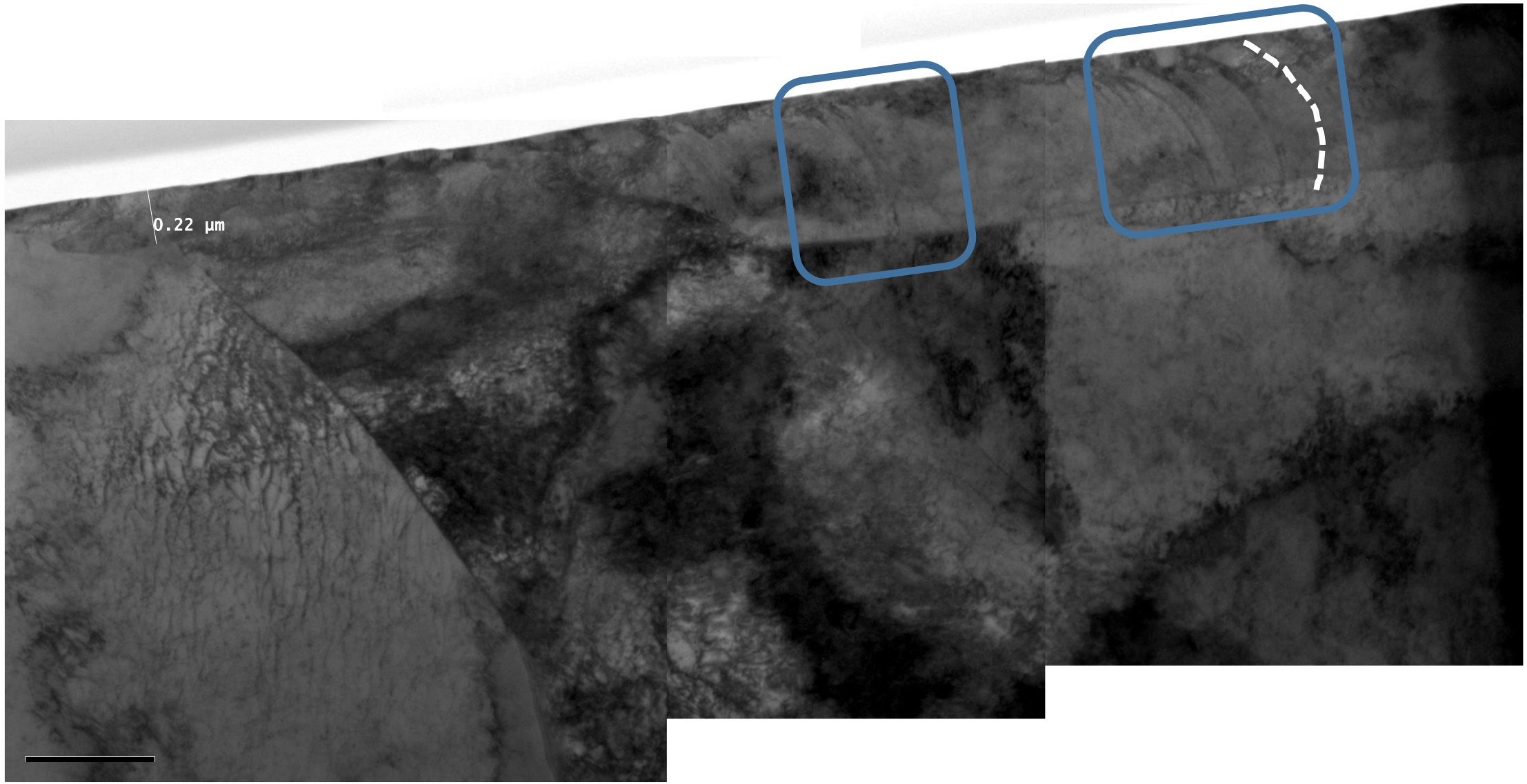


Hard Cu cathode@300 K – Reference Region: very similar extended twinned structures within the upmost ~ 500 nm

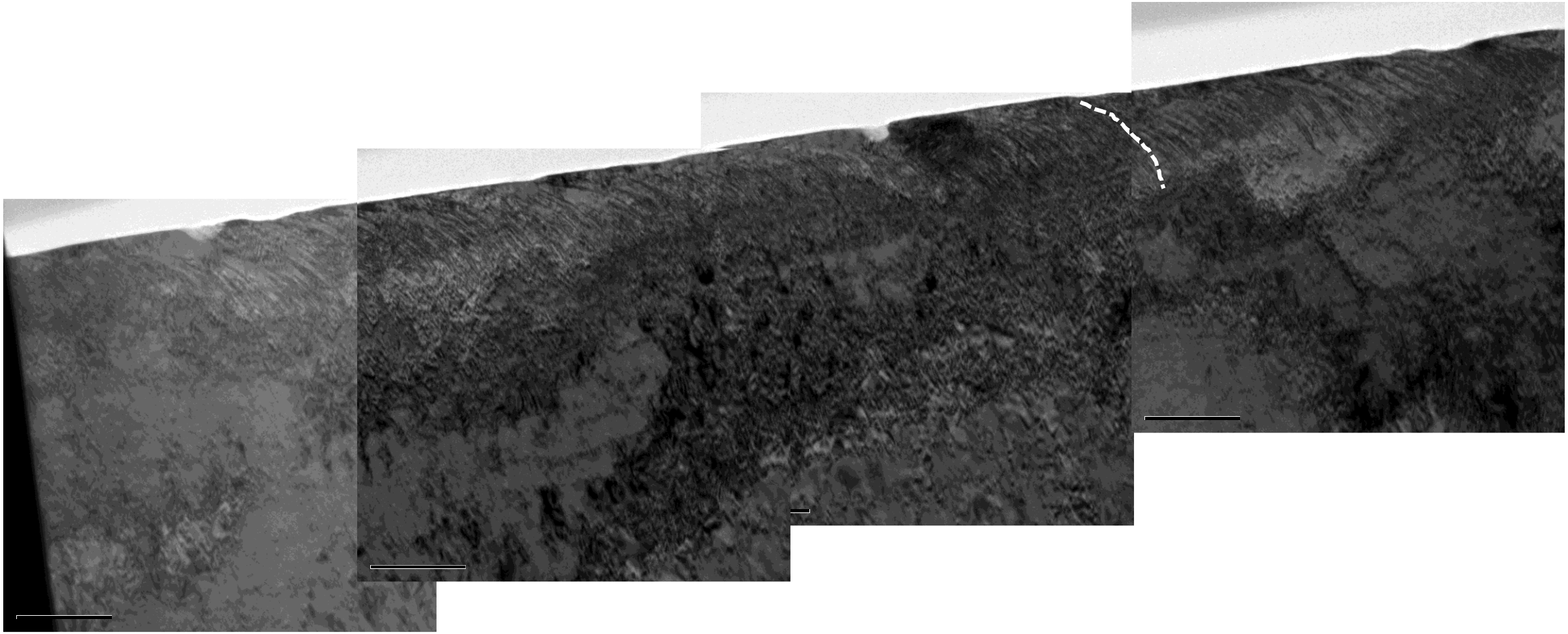




Hard Cu cathode@30 K – Reference Region: large blocks of twins within the upmost ~ 500nm

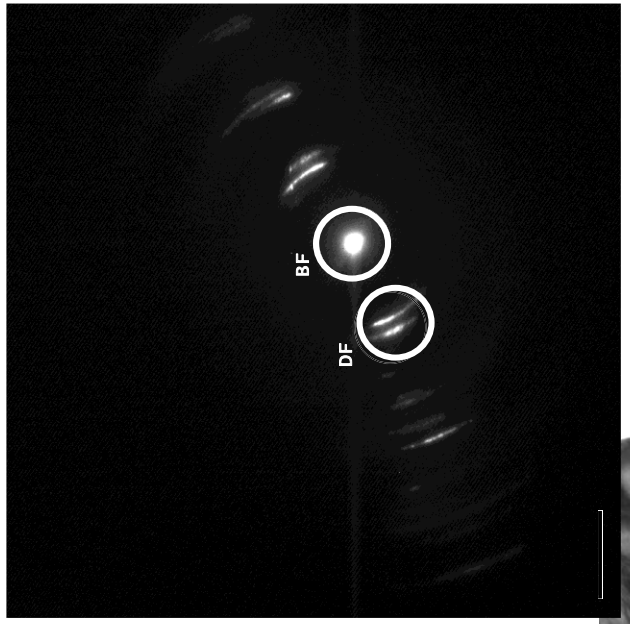


Hard Cu cathode@30 K – Reference Region: extended twinned structures within the upmost ~ 500nm

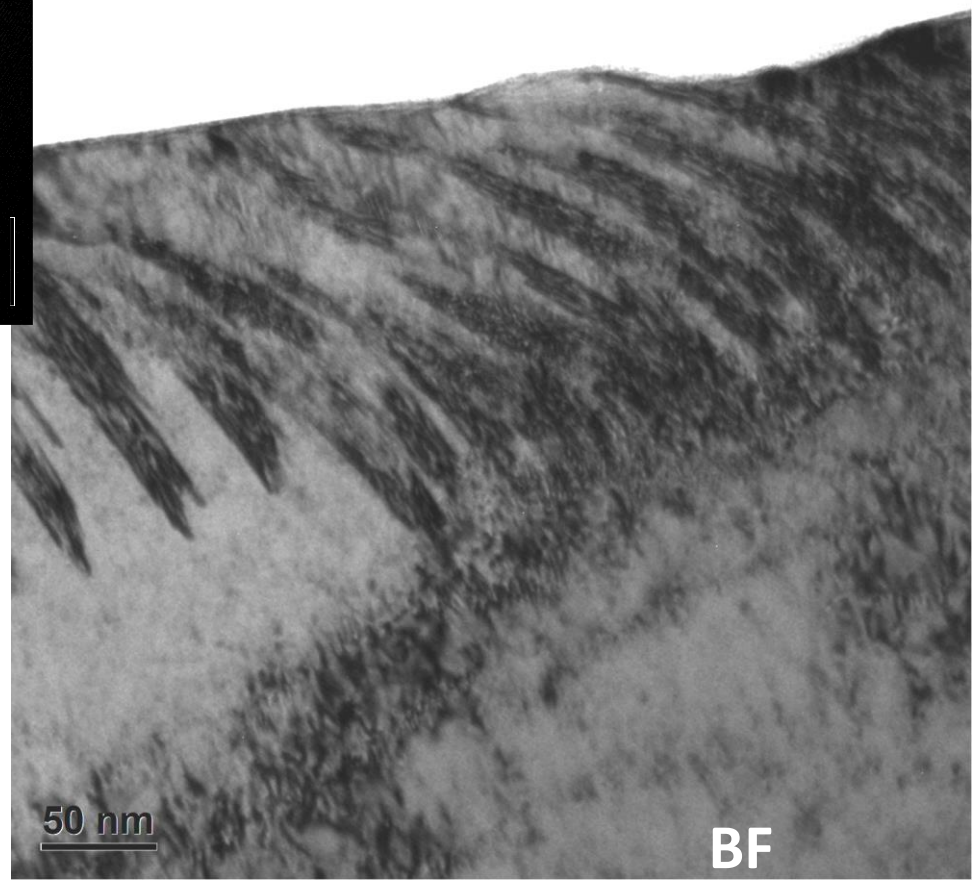


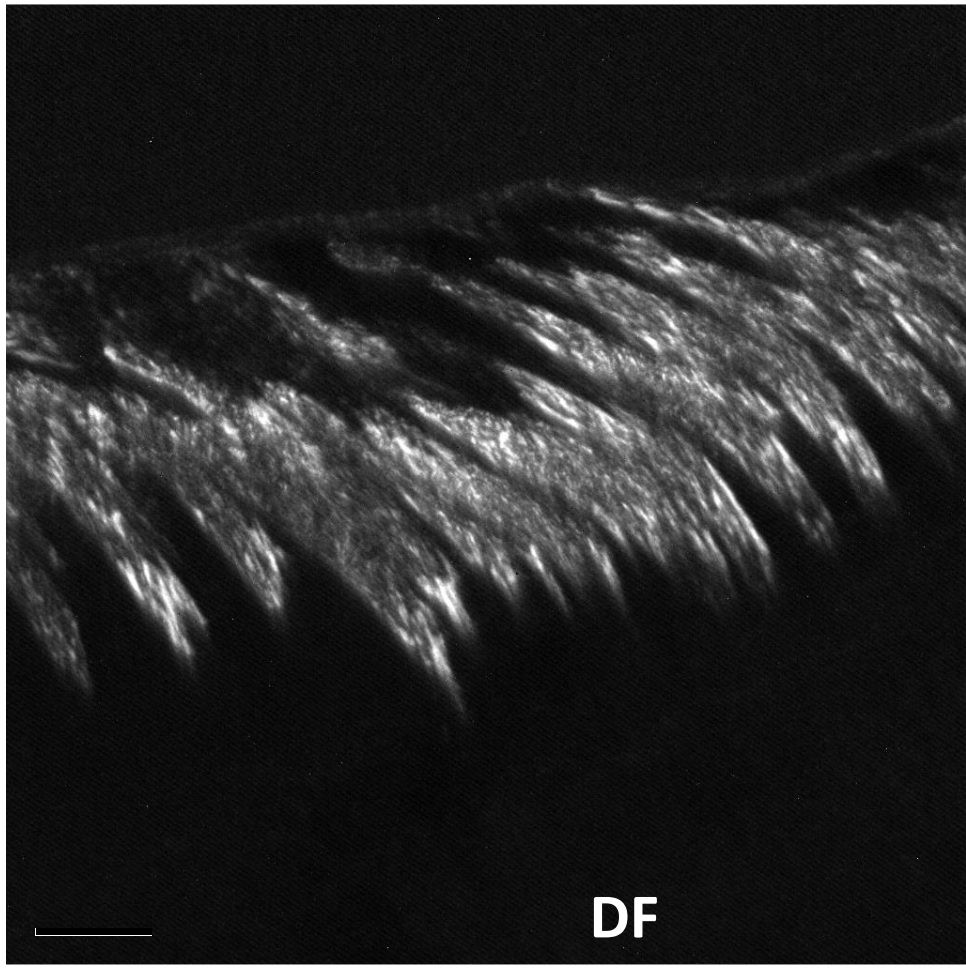
Lamella A1 (Ref region Nov 2022) – was extracted from a single grain

Hard Cu cathode@30 K – Reference Region: extended twinned structures within the upmost ~ 500nm



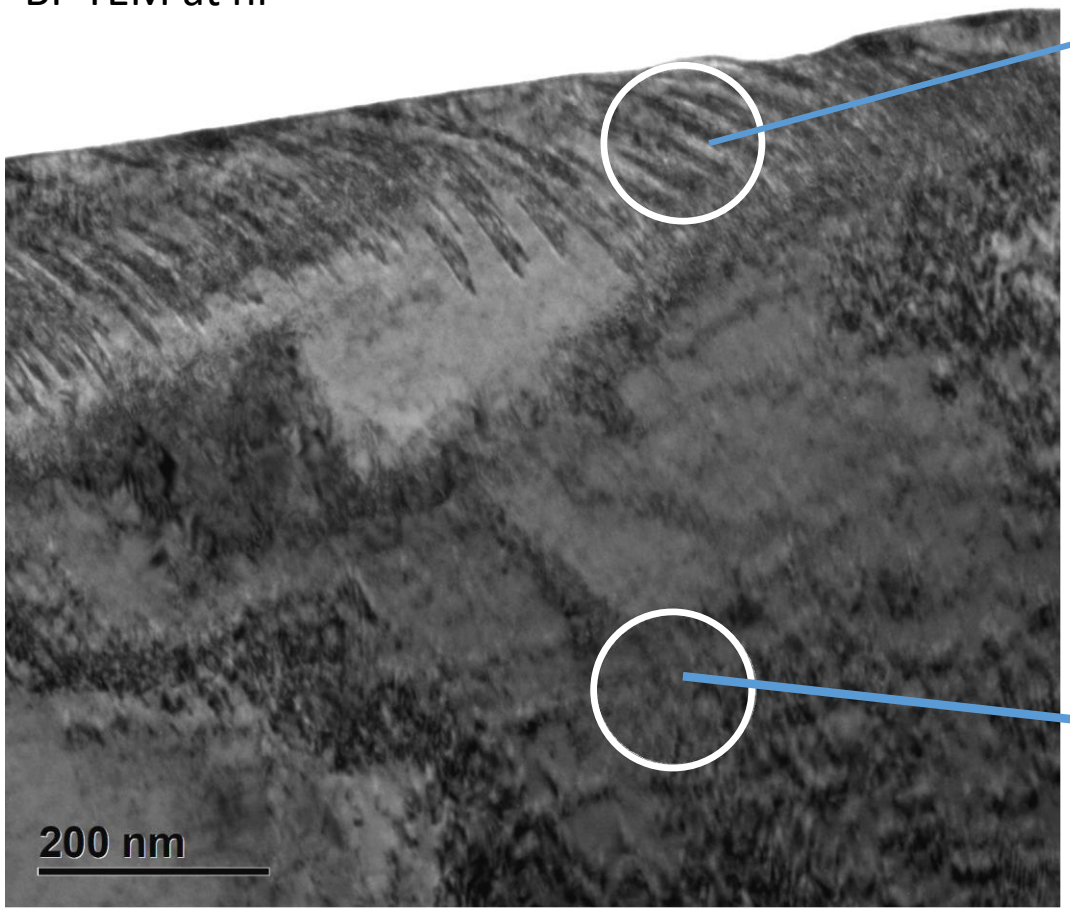
What are these rounded twin-like features?

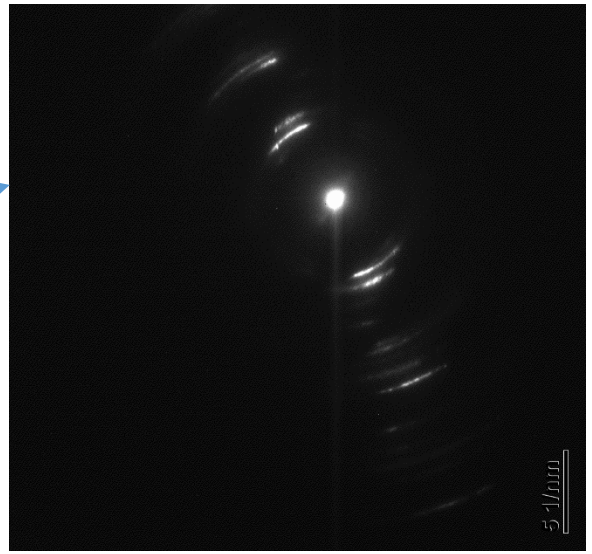




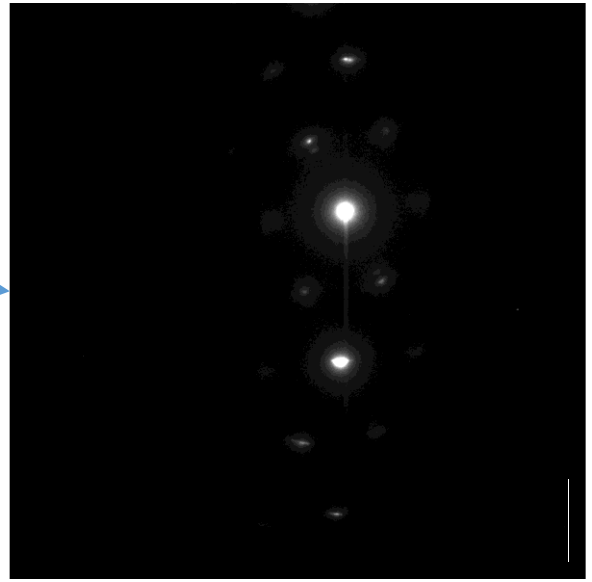
Hard Cu cathode@30 K – Reference Region: extended twinned structures within the upmost ~ 500nm

BF TEM at ni





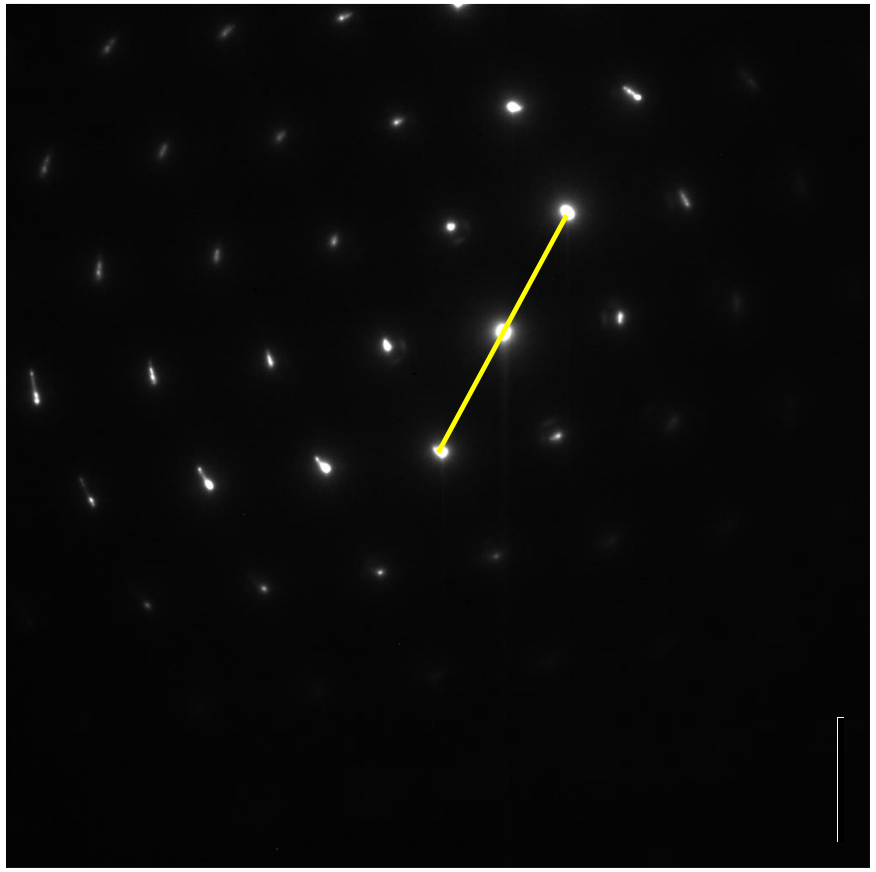
SAED acquired at a surface =
= a textured polycrystal?



SAED acquired below the surface (from bulk) = single crystal

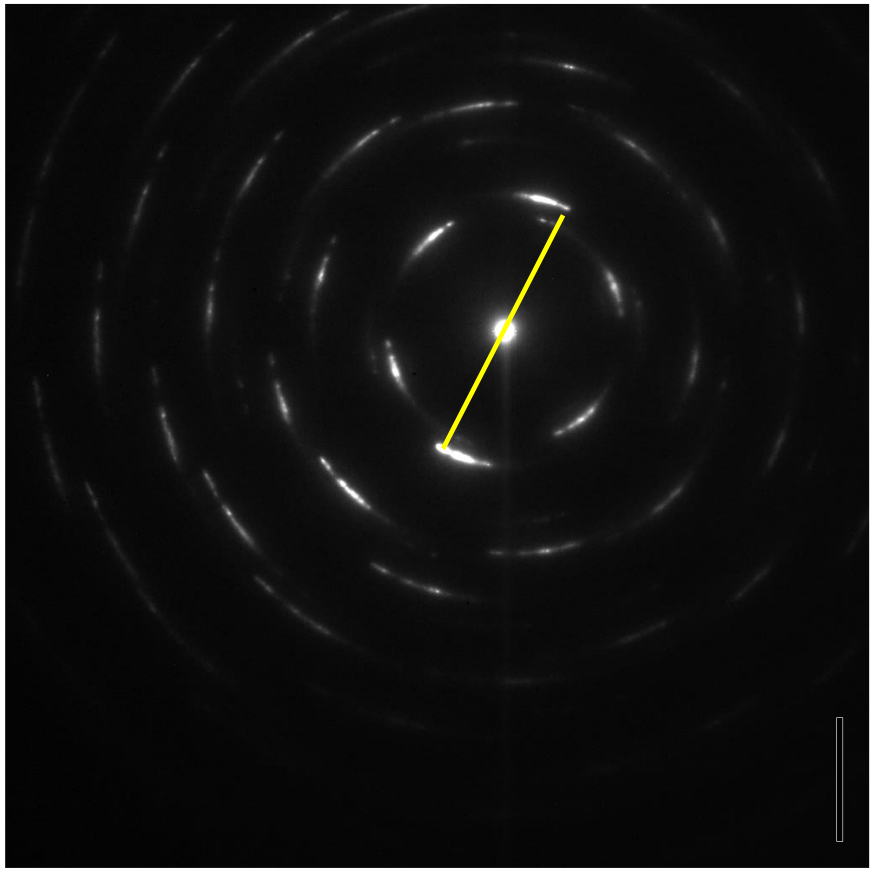
What are these rounded twin-like features?

Hard Cu cathode@30 K – Reference Region: extended twinned structures within the upmost ~ 500nm



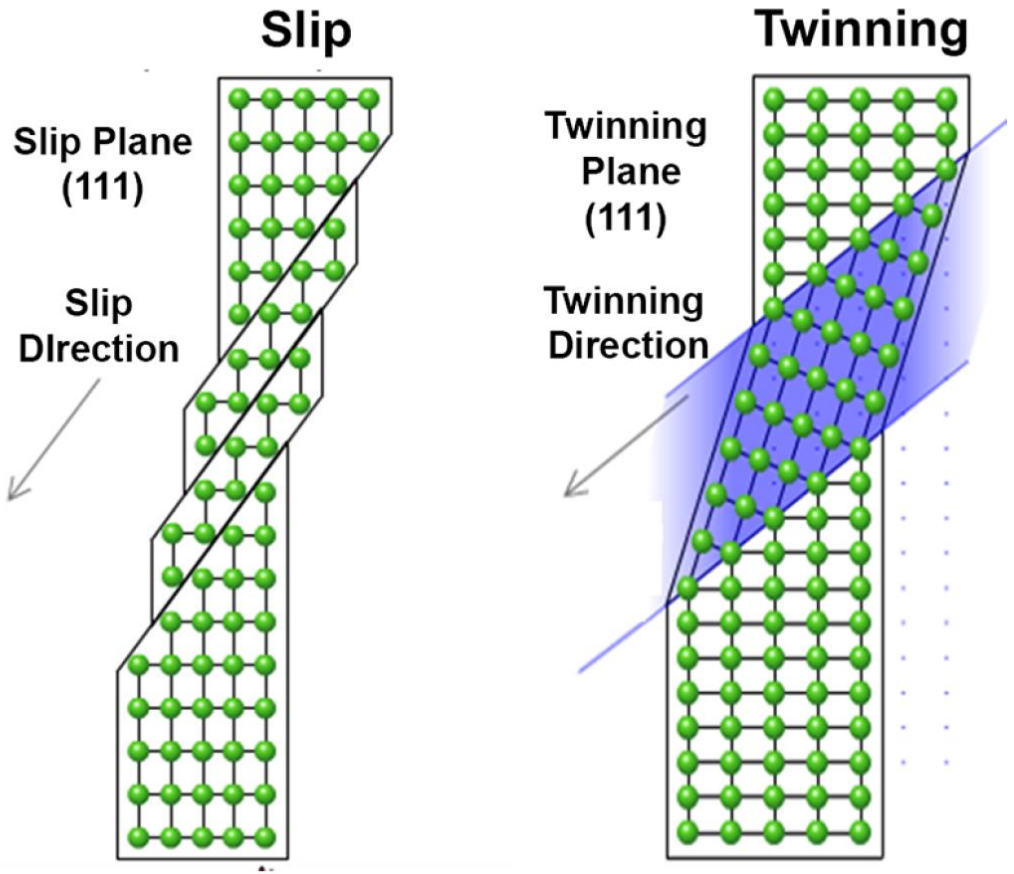
SAED acquired from a bulk :
Mother crystal is oriented almost in 110-type ZA

sample tilt a = -6 deg b= -2 deg



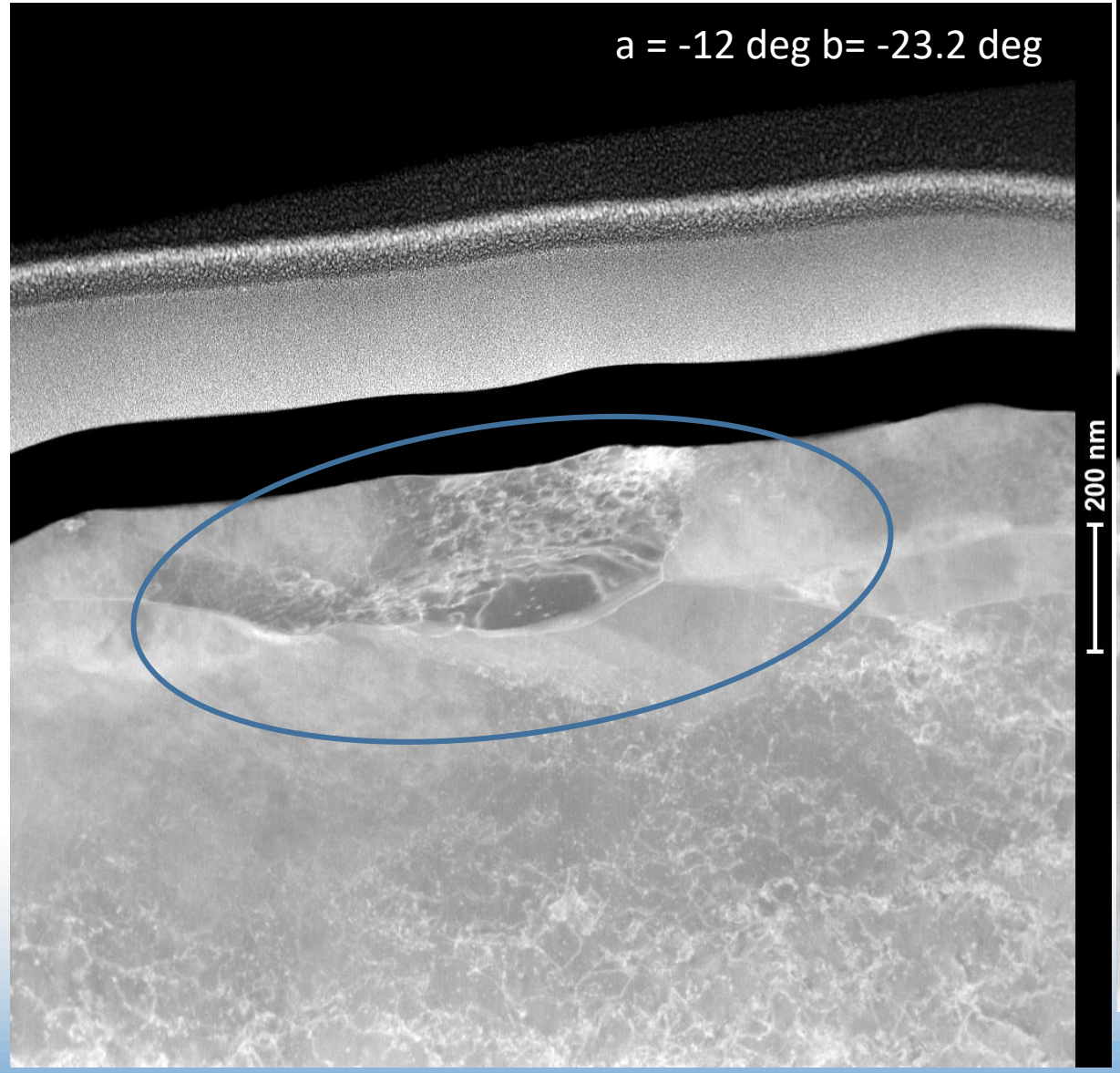
SAED acquired at a surface :
Orientation gradually deviates from ZA of a mother crystal: DOTs are transformed into ARCs!
At a pure twinning we would observe a line of less intense dots behind the main one (along a straight line)
Arcing indicates 1) gradual misorientation=gradual rotation/tilting, and 2) grain refinement (defragmentation), considering the same diffracting volume (=the same aperture size)

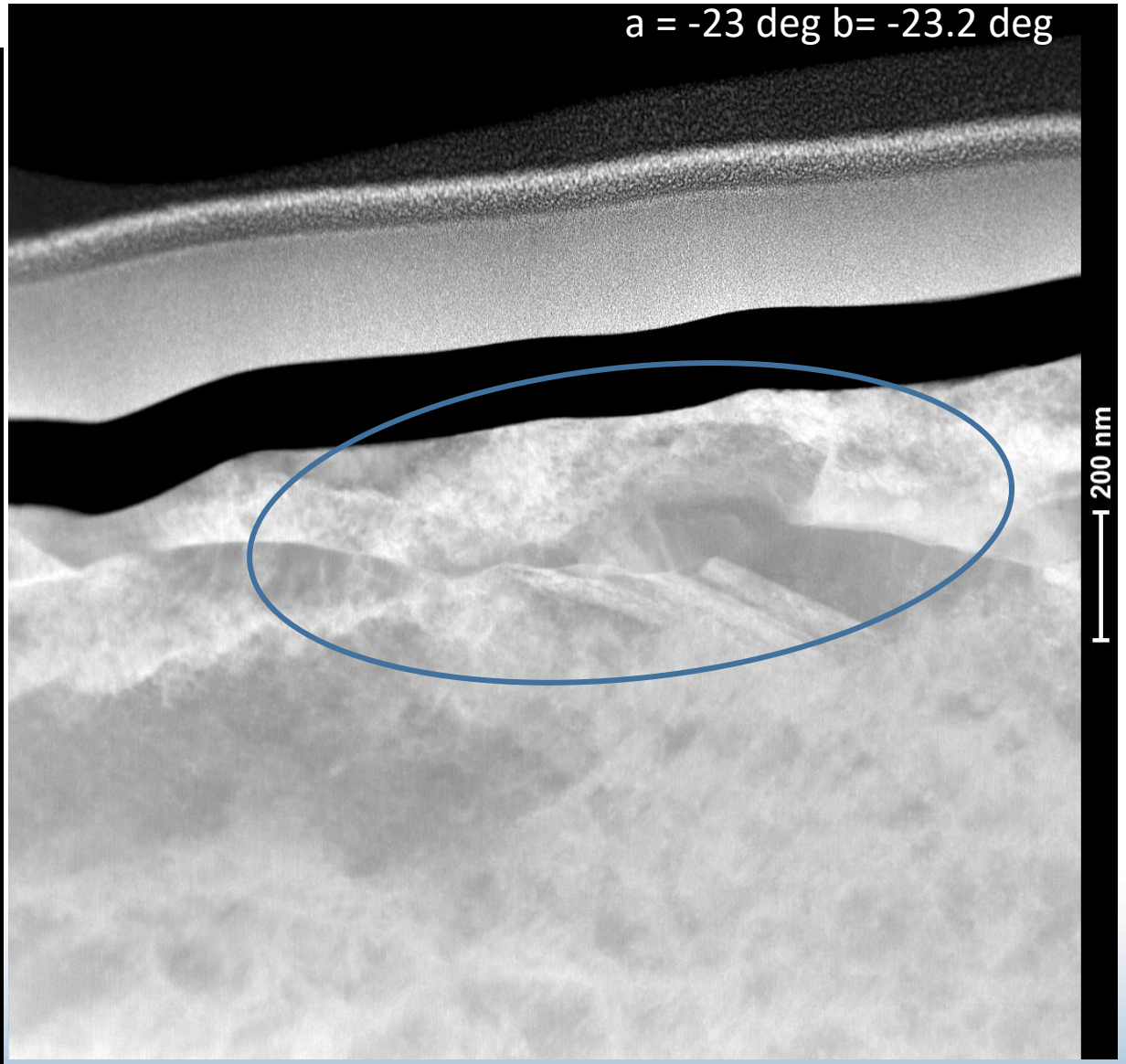
Hard Cu – Reference Region – surface hardening/deformation via surface grain defragmentation and gradual mis-orientation (shuffling)



Deformation Shuffling = Slip@InPlane Rotation + Twinning

Hard Cu – Field– Exposed Regions: the extended twinned structures are absent; small twin blocks are still observed, arcing on SAEDs is almost absent

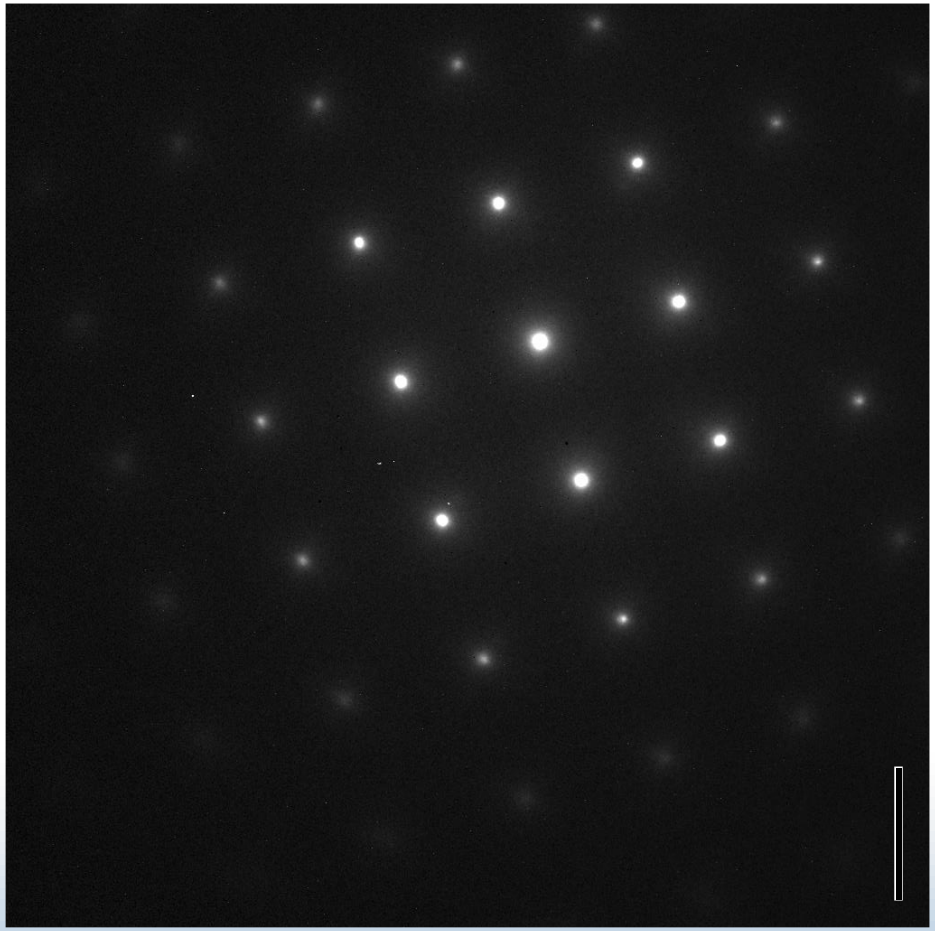




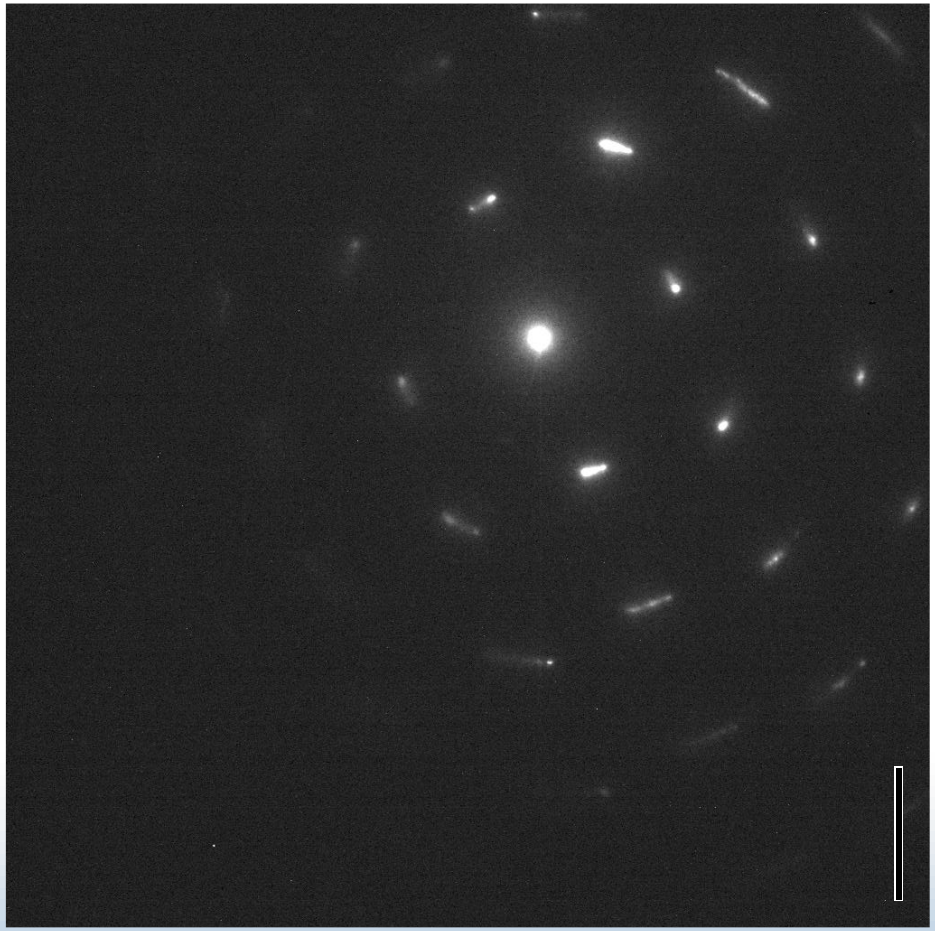
Hard Cu – Field– Exposed Region: arcing on SAEDs is either absent or weakly expressed

Cathode@30K , TEM, SAED

Bulk

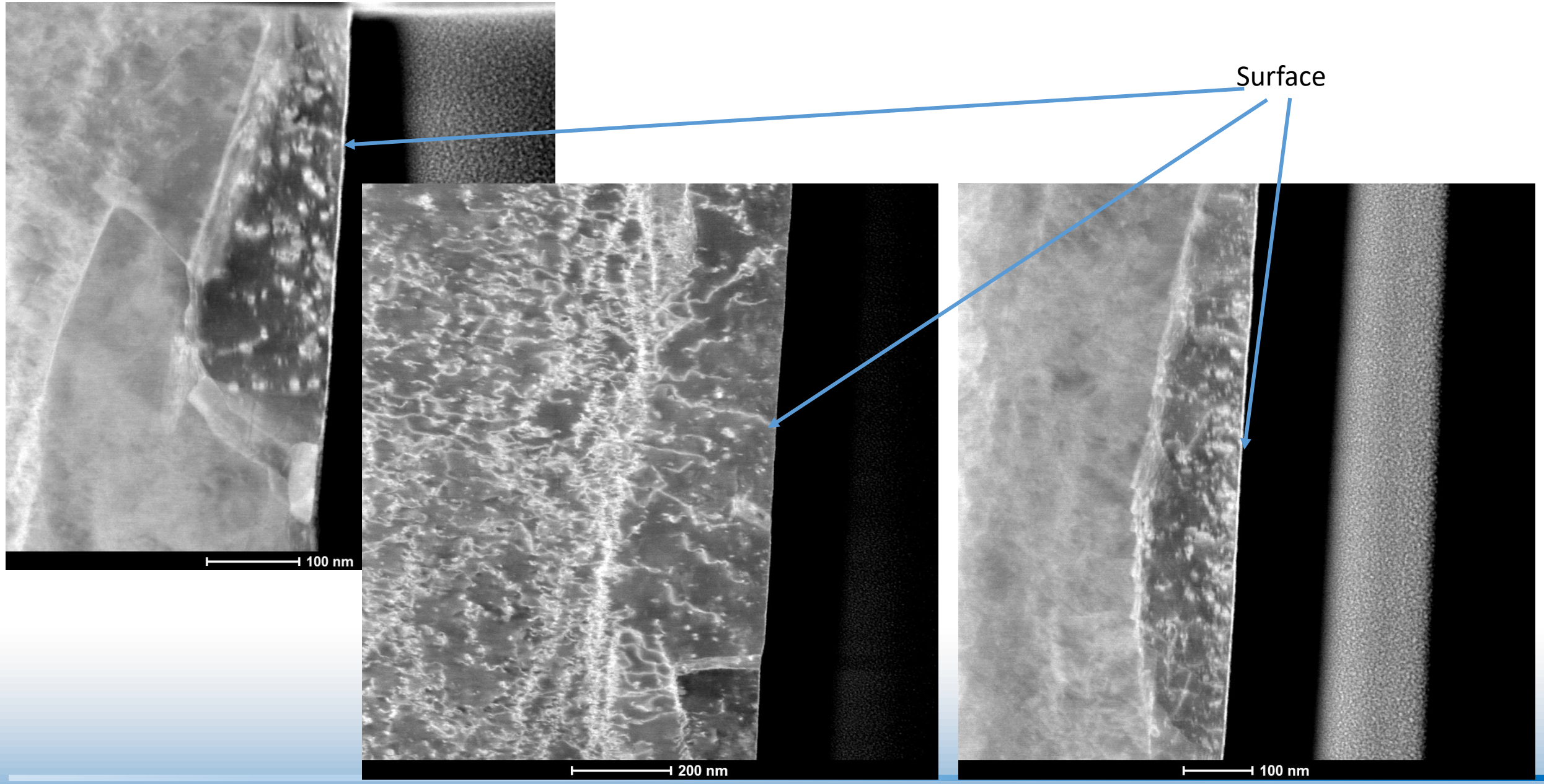


Surface



ZA<100> a = 7.1 deg b= 12.2 deg

Hard Cu – Field – Exposed Regions of Cathode@30K: dislocation depletion in the surface grains?



What is affected by cooling ?

- Shape, topography and size of BDs
- Maybe: dislocation movement/reactions causing depletion

What happens in a subsurface of FE regions?

- Structural relaxation of annealing-type, i.e. a global decrease in extent of mis-orientation (the effect is quite the same for the electrodes tested at 30 and 300 K)

What could be the reason/mechanism of the observed structural changes?

- A heat release in due course of thermal runaway/BDs
- or a lattice defects-related response to Field Exposure (detwinning, dislocation interaction with GBs at any T, dislocation annihilation near the surface primarily under cooling) ?