Microstructural features of pre- and post-BD RF- tested samples: facts and questions

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CLIC, CERN

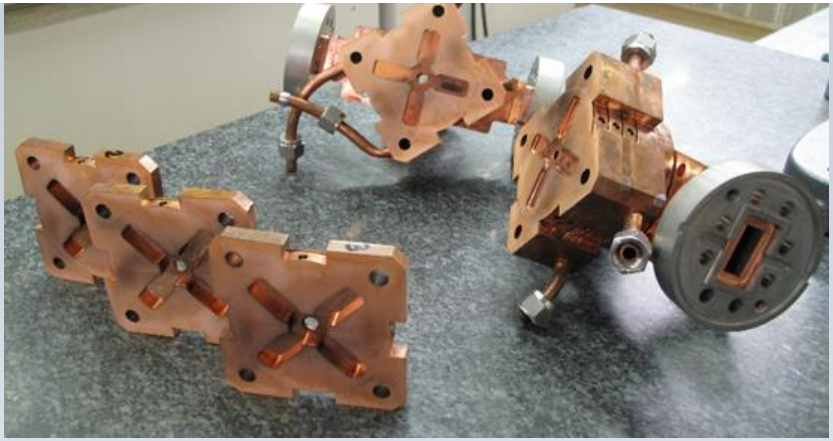
MeVARC-5, Lapland

2-4 September, 2015

האוניברסיטה העברית בירושלים
The Hebrew University of Jerusalem

EDM wire cutting machine at CERN workshop:

Structure just after cutting:



Structure ready for SEM after degreasing:

US of the cells:

Iris #4 Iris #5 Iris #6

Iris #22

DS of the cells:

Iris #4 Iris #5 Iris #6 Iris #23

The samples:

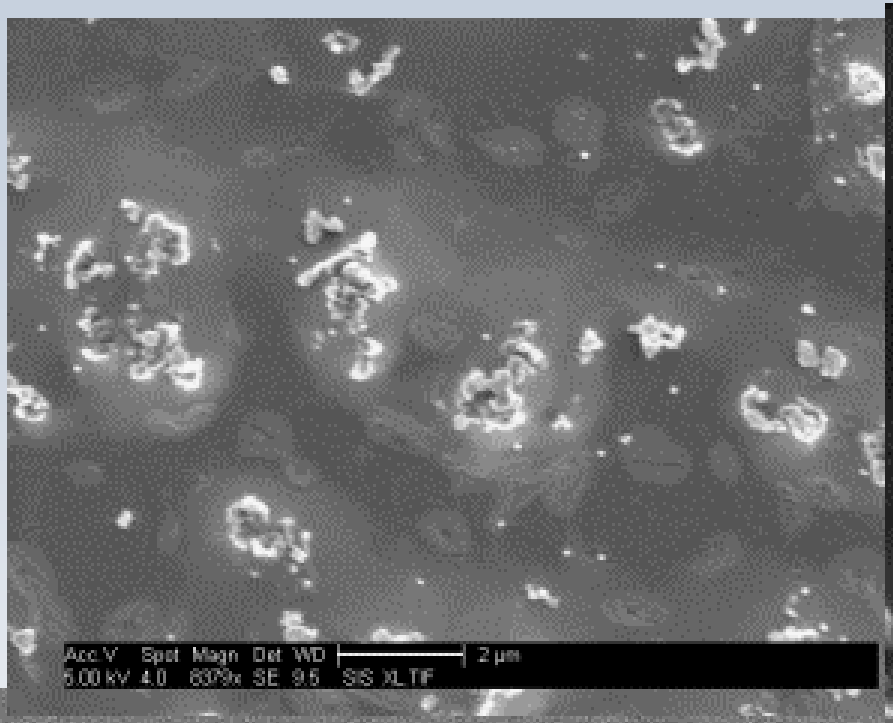
- Reference disk
- RF tested disk#22
- RF tested disk#23

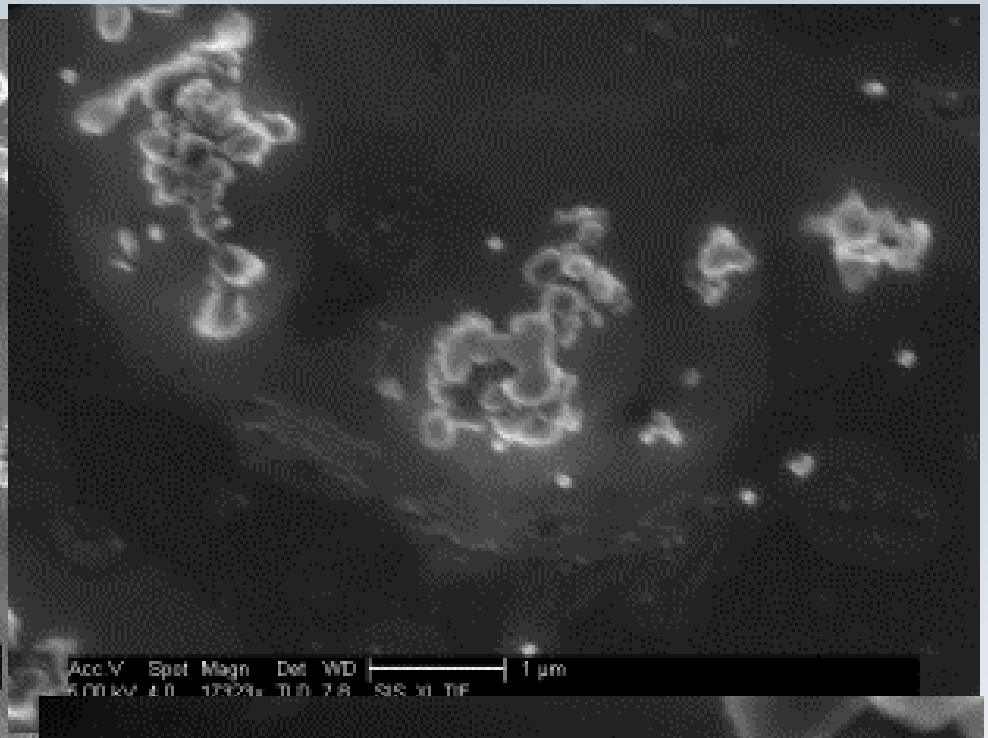
The task:

- To identify the microstructural features related to pre-BD structure

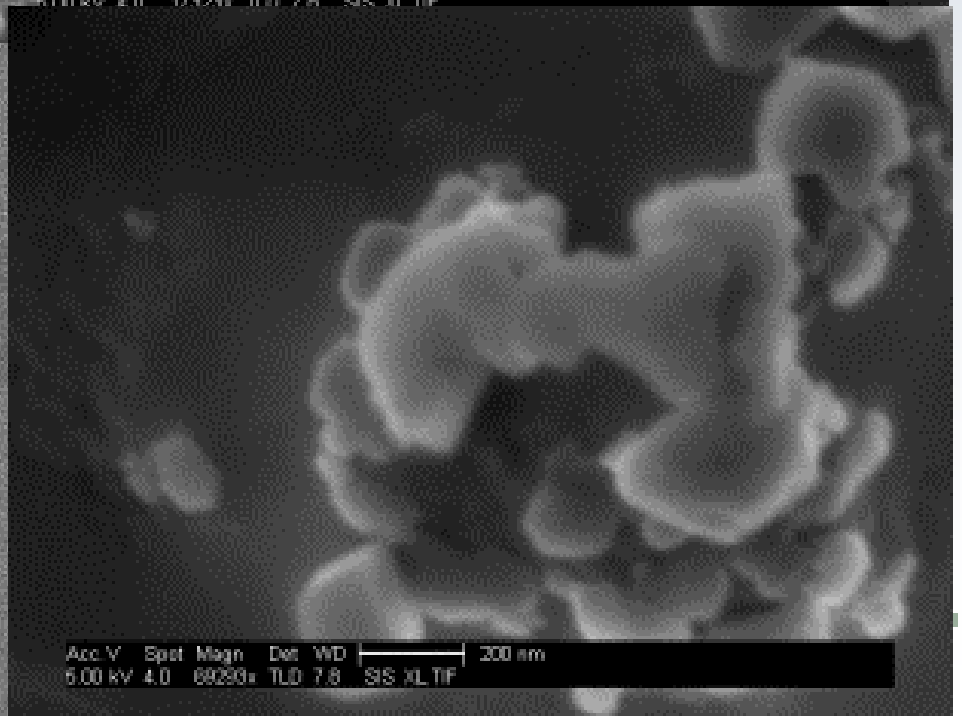
- - - to distinguish between post-production and post-RF-test features

- - - to identify the defect structure of RF-tested samples (dislocations, clusters of vacancies, etc)

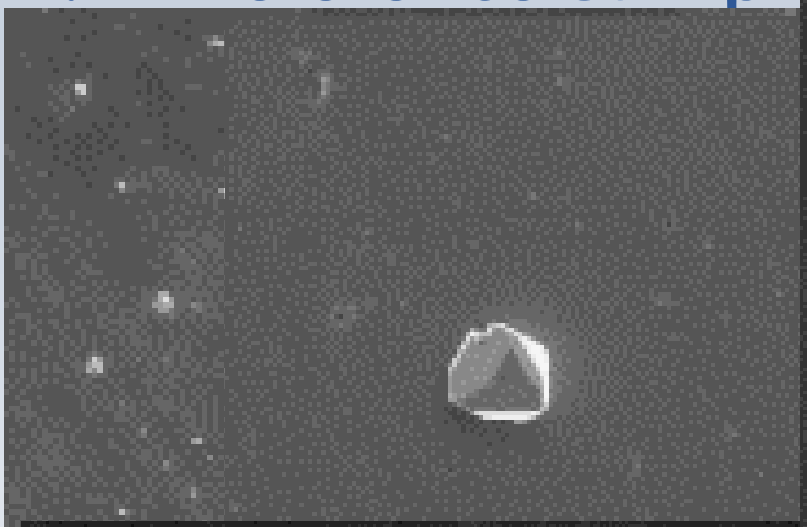

Acc.V Spot Magn Det WD 2 µm
5.00 kV 4.0 6979x SE 9.5 SIS XL.TIF

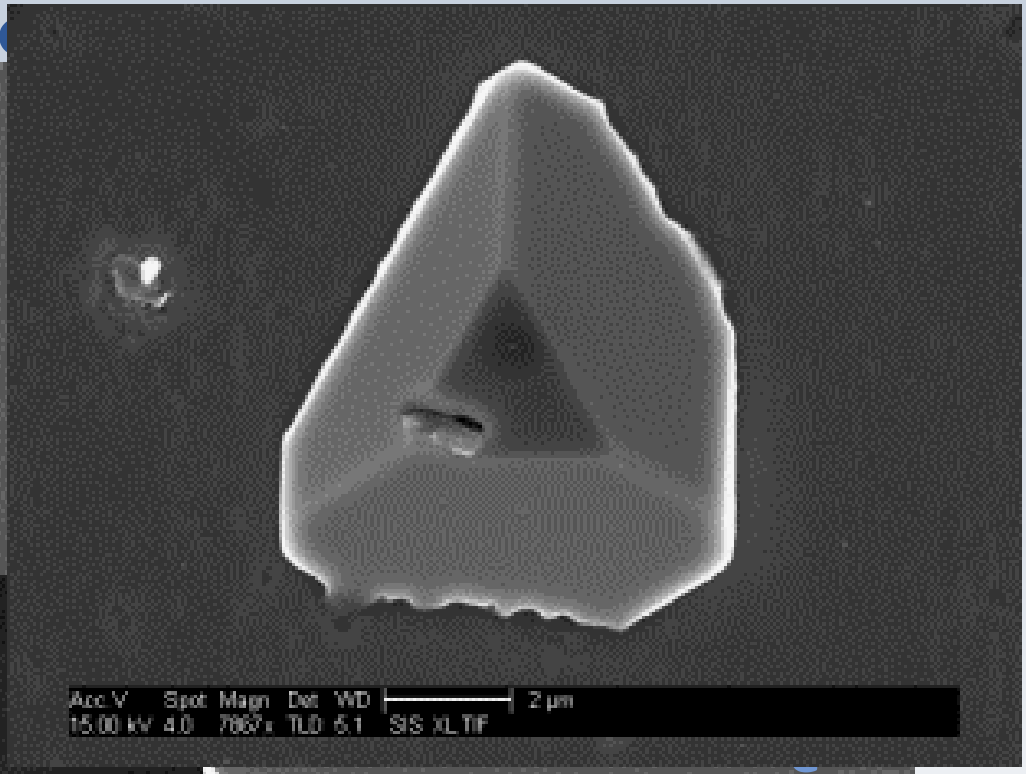

Acc.V Spot Magn Det WD 1 µm

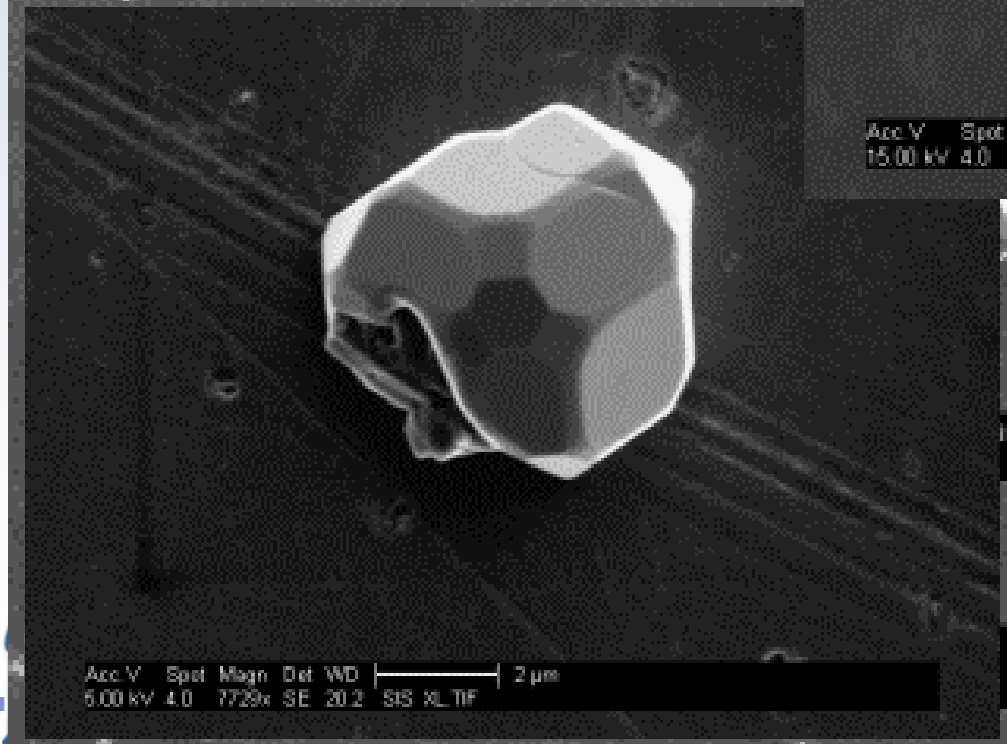

Acc.V Spot Magn Det WD 200 µm
5.00 kV 4.0 131x SE 9.5 SIS XL.TIF


Acc.V Spot Magn Det WD 200 nm
5.00 kV 4.0 69290x TLD 7.8 SIS XL.TIF

I. A Reference Sampl

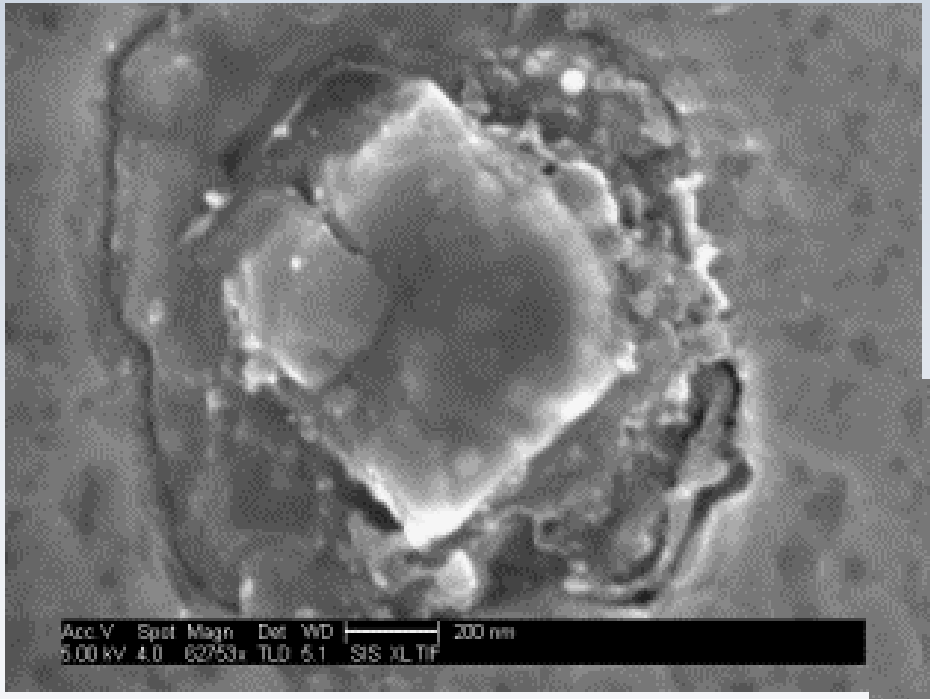


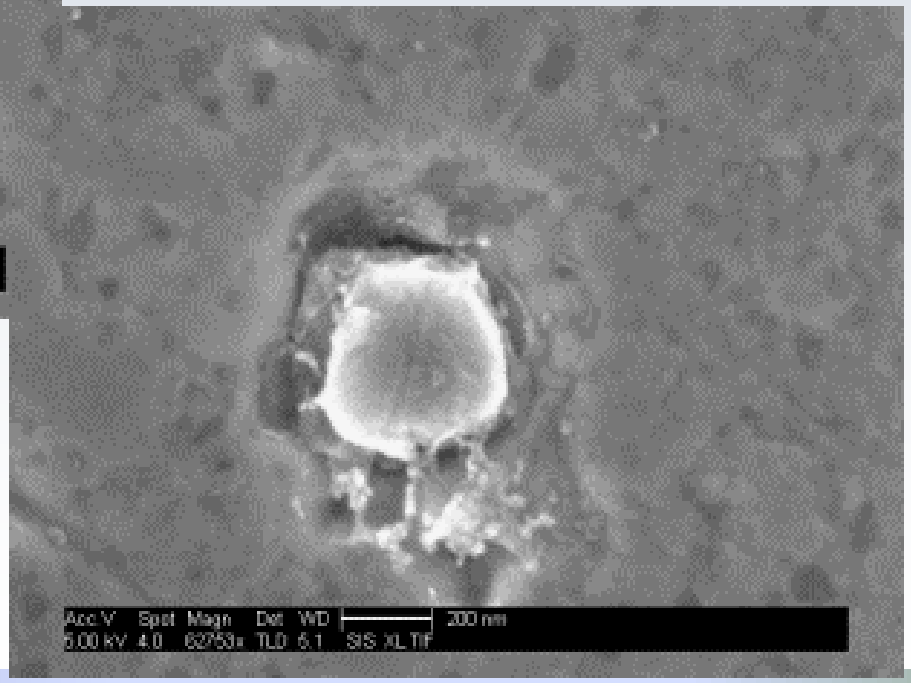


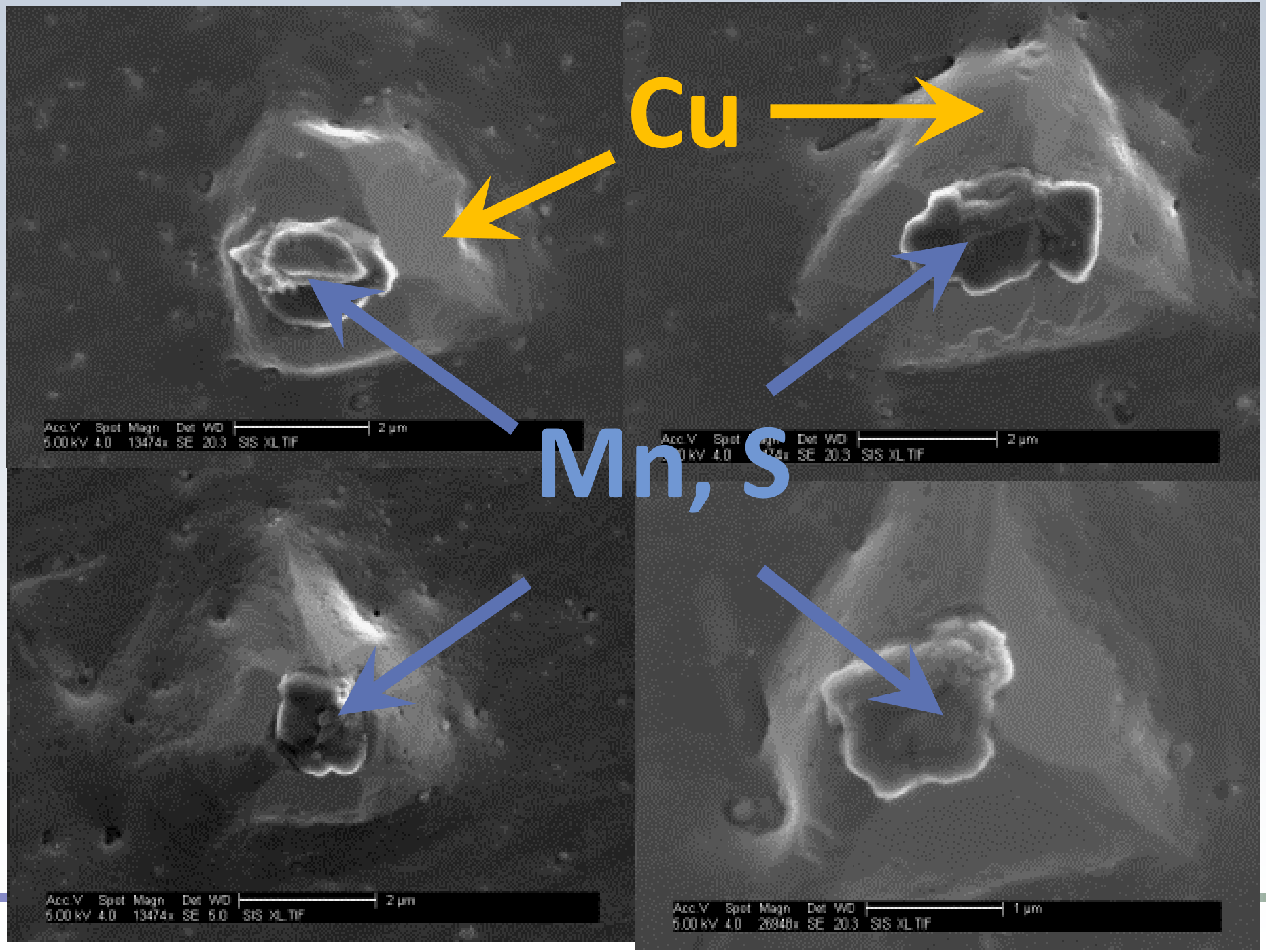


I. A Reference Sample

- fine non-conductive particles - oxides





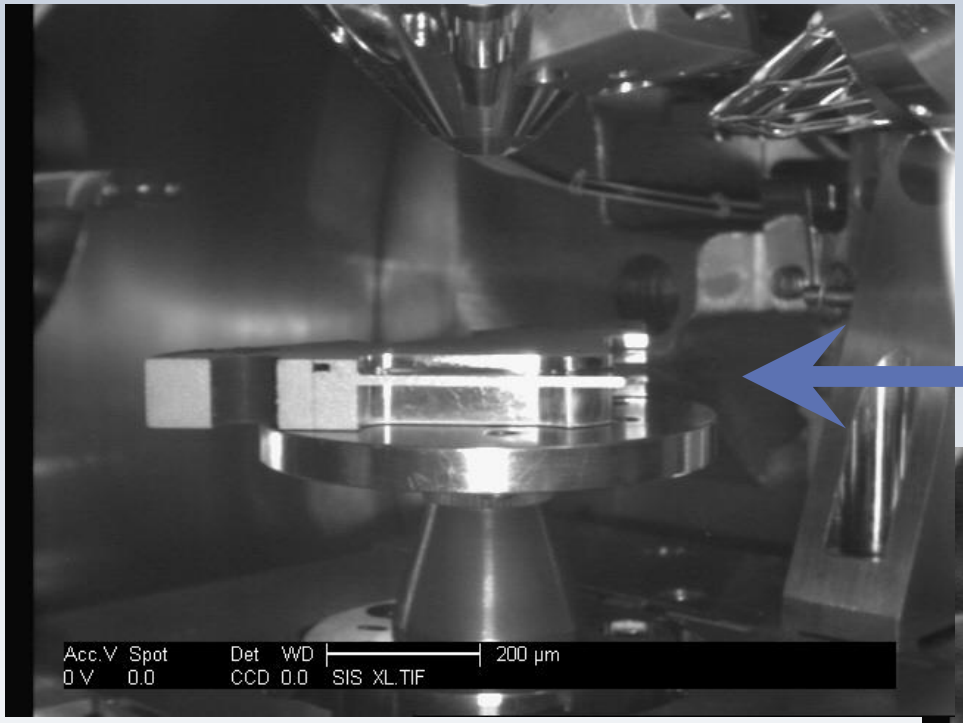

Cu
Mn, S
Acc.V Spot Magn Det WD 2 µm
5.00 kV 4.0 13474x SE 20.3 SIS XL.TIF
Acc.V Spot Magn Det WD 2 µm
5.00 kV 4.0 13474x SE 5.0 SIS XL.TIF
Acc.V Spot Magn Det WD 1 µm
5.00 kV 4.0 26948x SE 20.3 SIS XL.TIF

I. A Reference Sample

- ✓ A reference sample was not suitable to answer the task questions
- ✓ If not occasional, this is a problem of raw OFE Cu quality or uniformity

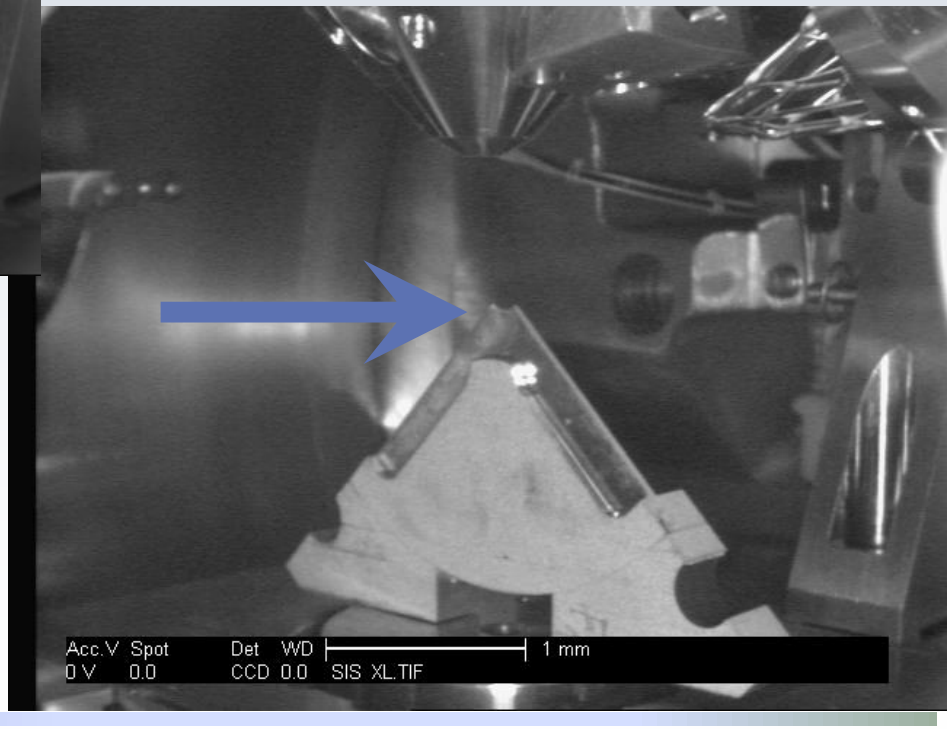
! After-production - Cu is extremely soft =
= almost fully relaxed closed to equilibrium microstructure = low density of dislocations

II. RF-tested disk#22



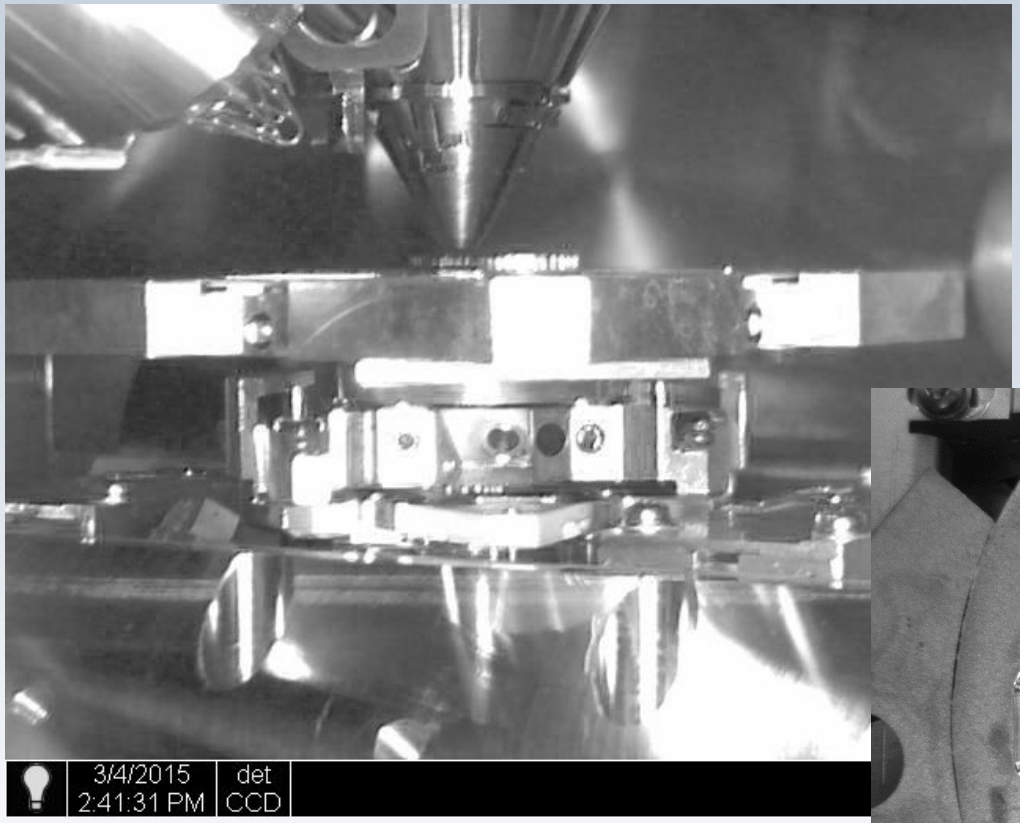
Nothing resembling BDs

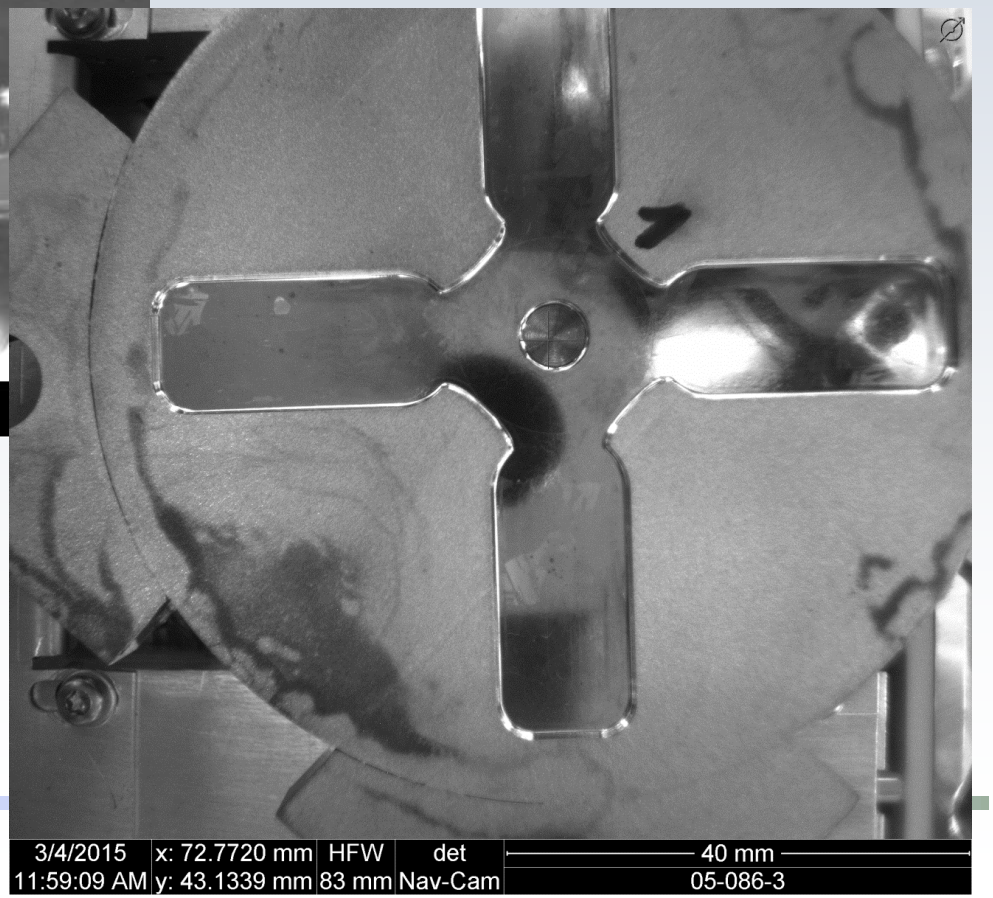
We analyzed iris



RF-tested disks MUST be analyzed as they are

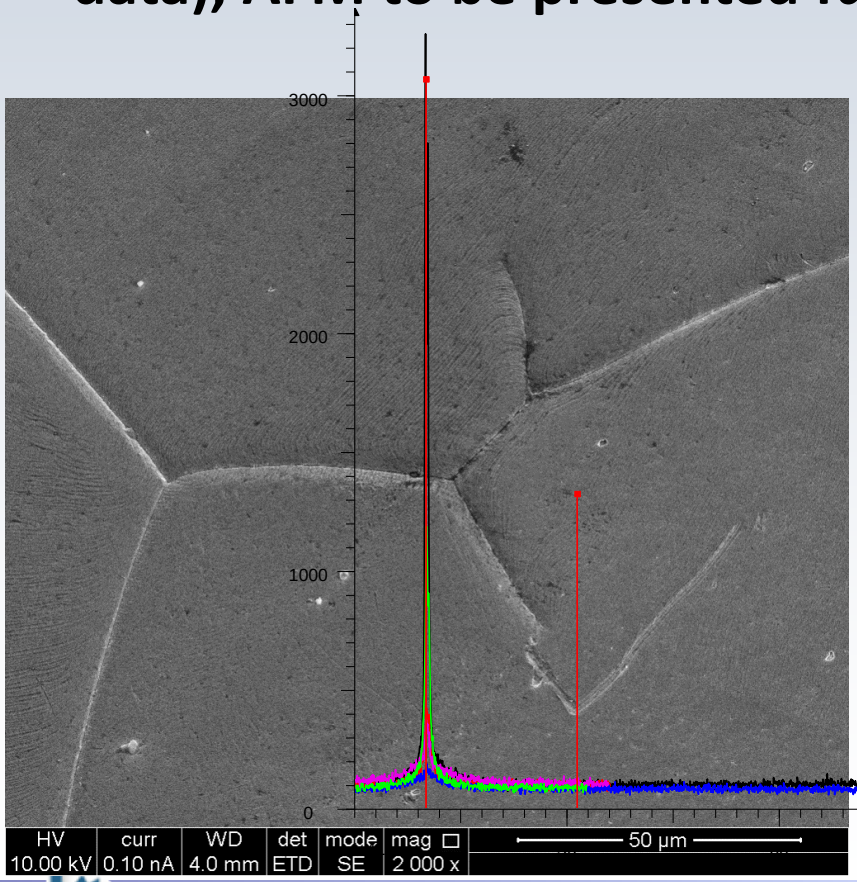
III. RF-tested disk#23

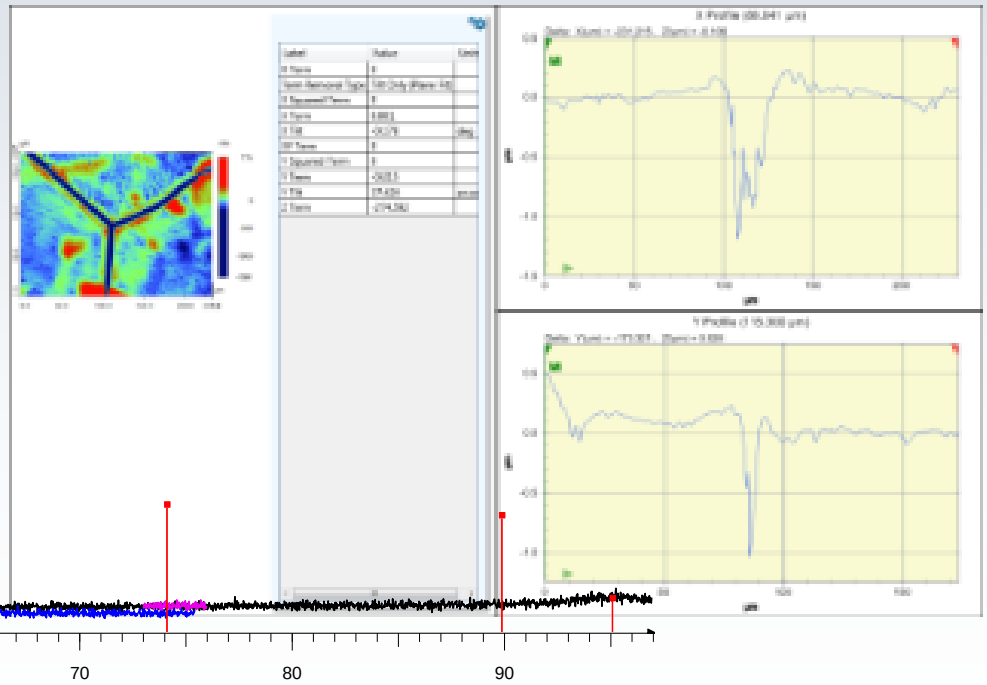




III. RF-tested disk#23

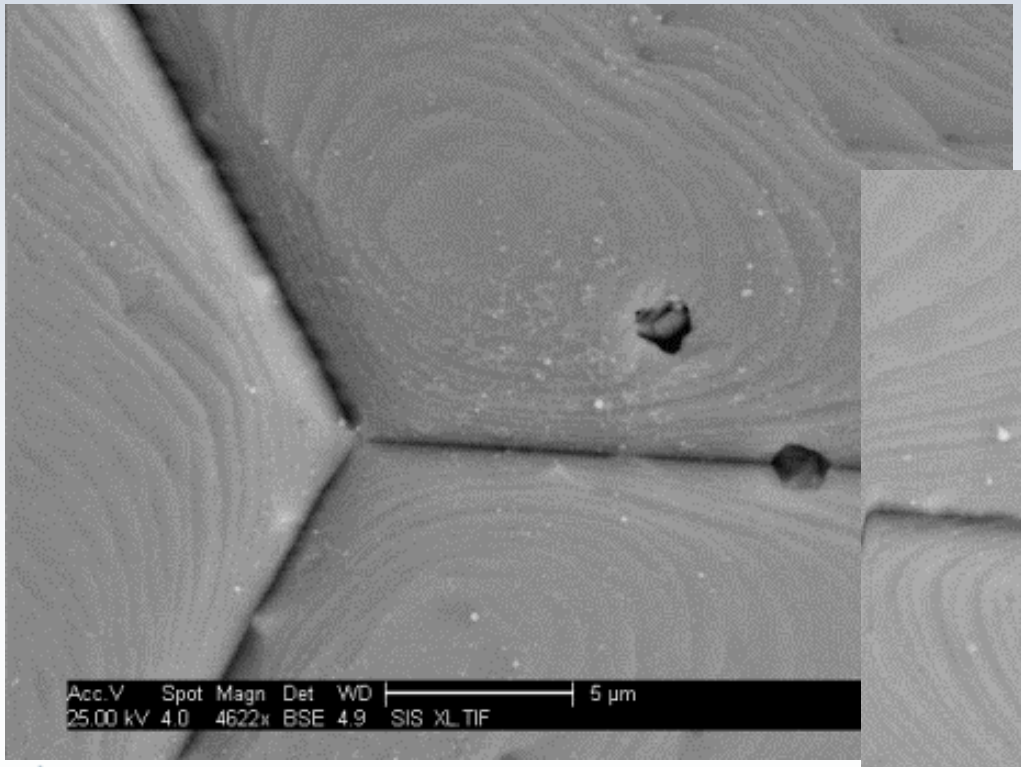
- Huge submillimeter grains
- The majority of the grains are <111>- oriented (XRD data)
- Micron-depth grain boundary grooves (optical profylometer data), AFM to be presented further by Ayelet)

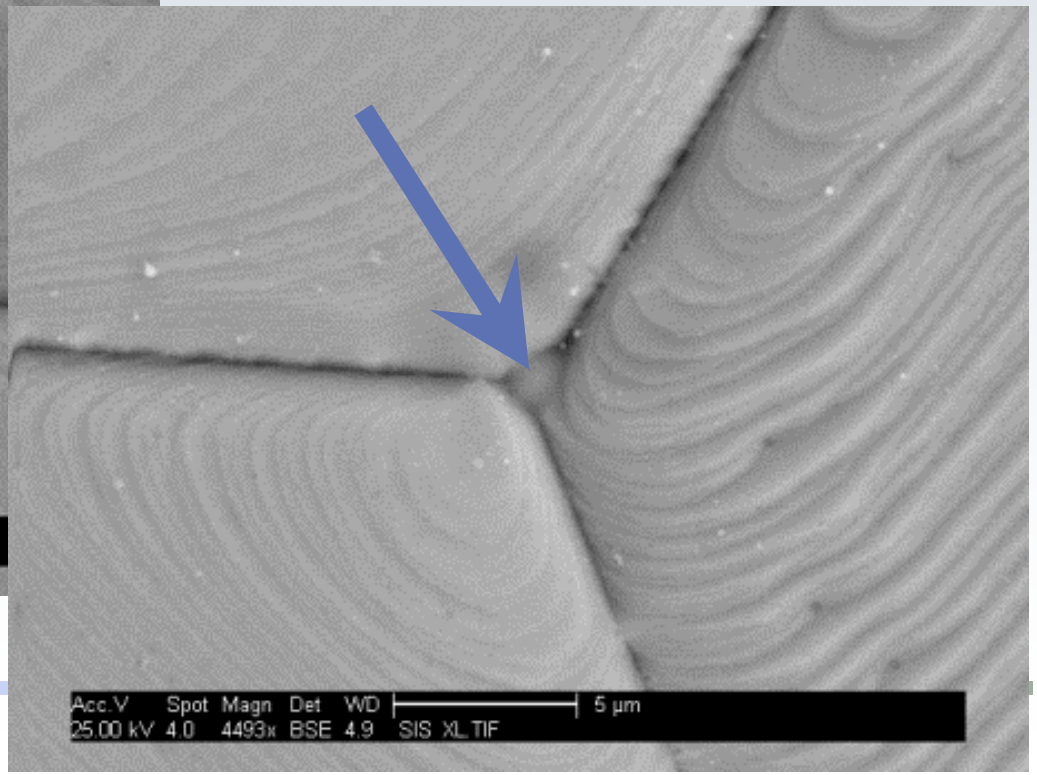




III. RF-tested disk#23

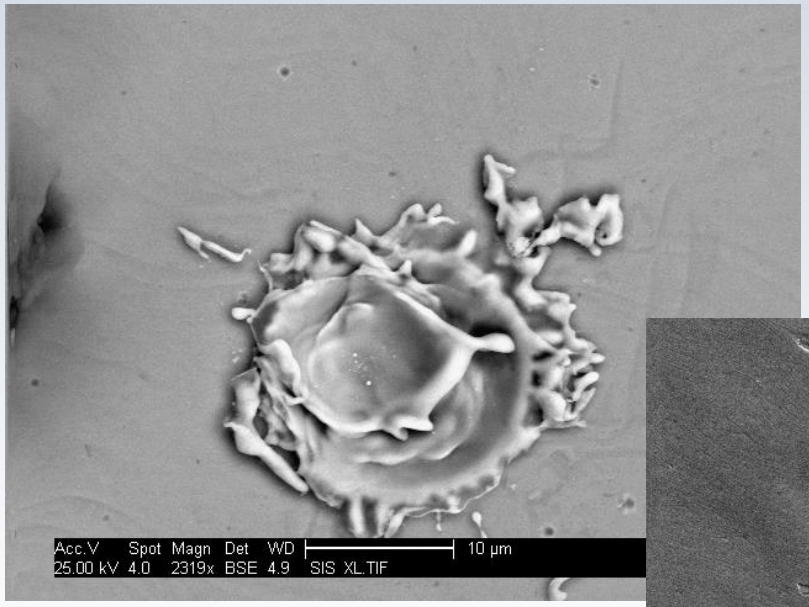
- Facetted grain surface (terraces of 5-50 nm height)
- Equilibrium grain boundary structure: 120 deg triple junctions
- Non-metallic inclusions

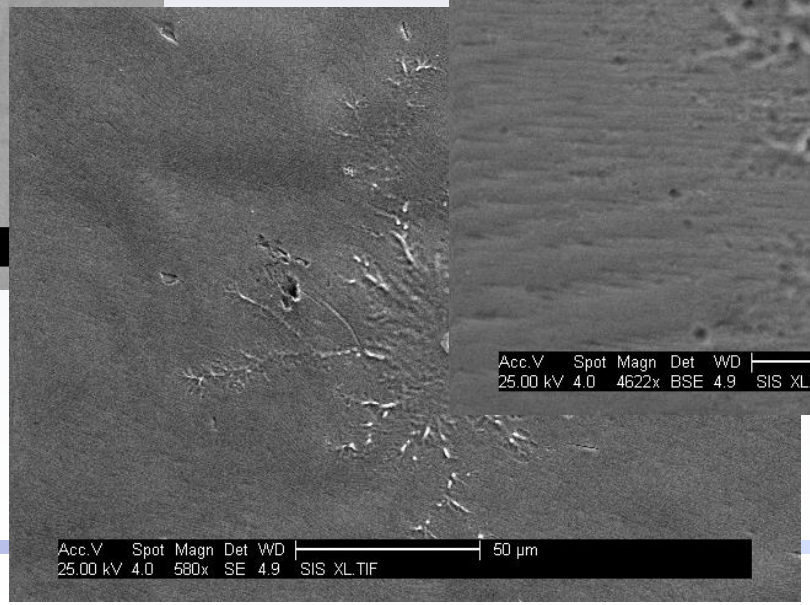


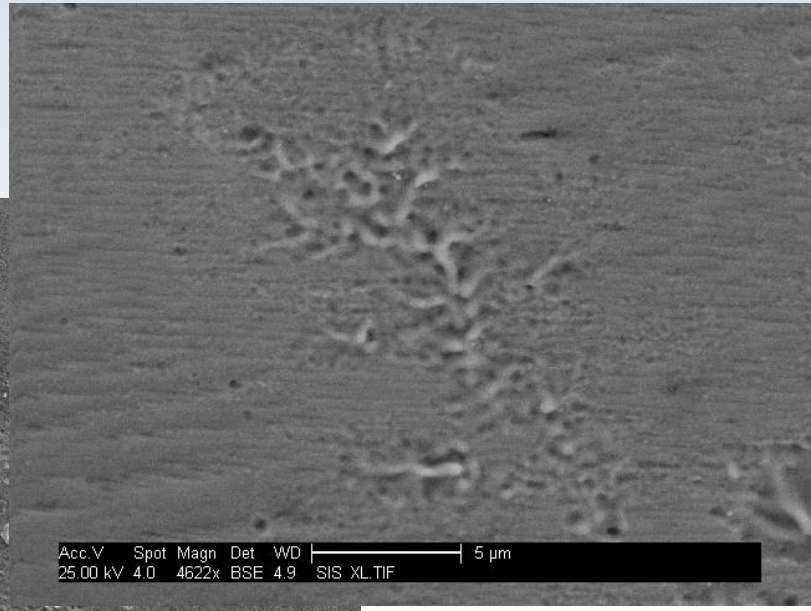


III. RF-tested disk#23

- Spark sites/splashes similar to BDs observed in DC-tested samples
- “New features” - dendrite-like and symmetrical
- Holes of various sizes in grain bodies and grain boundaries

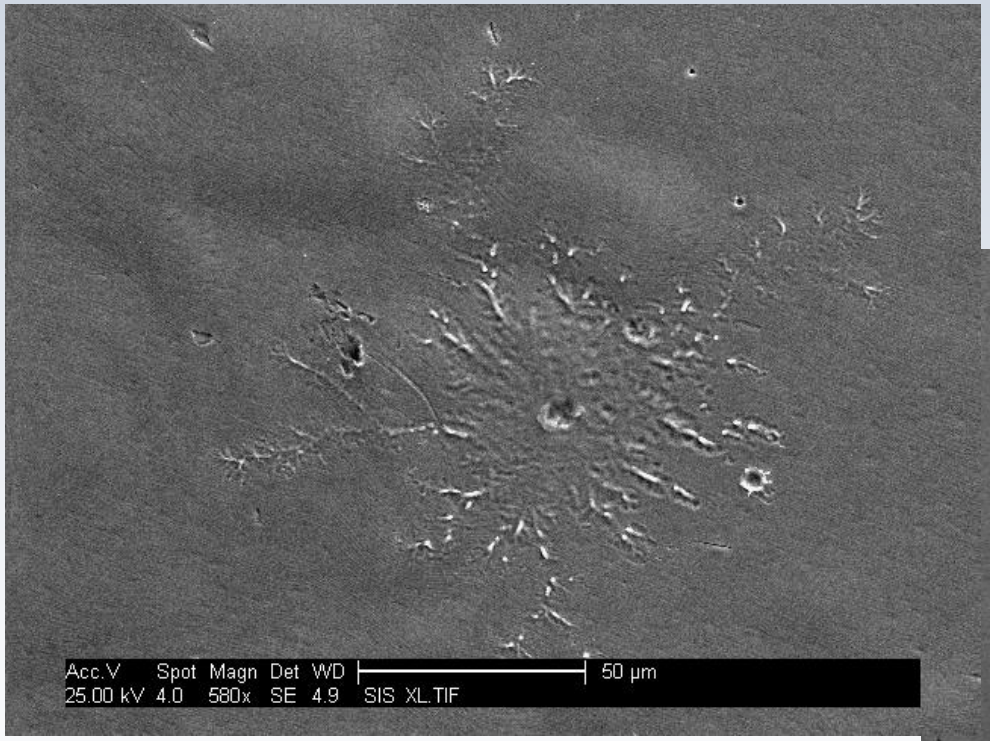


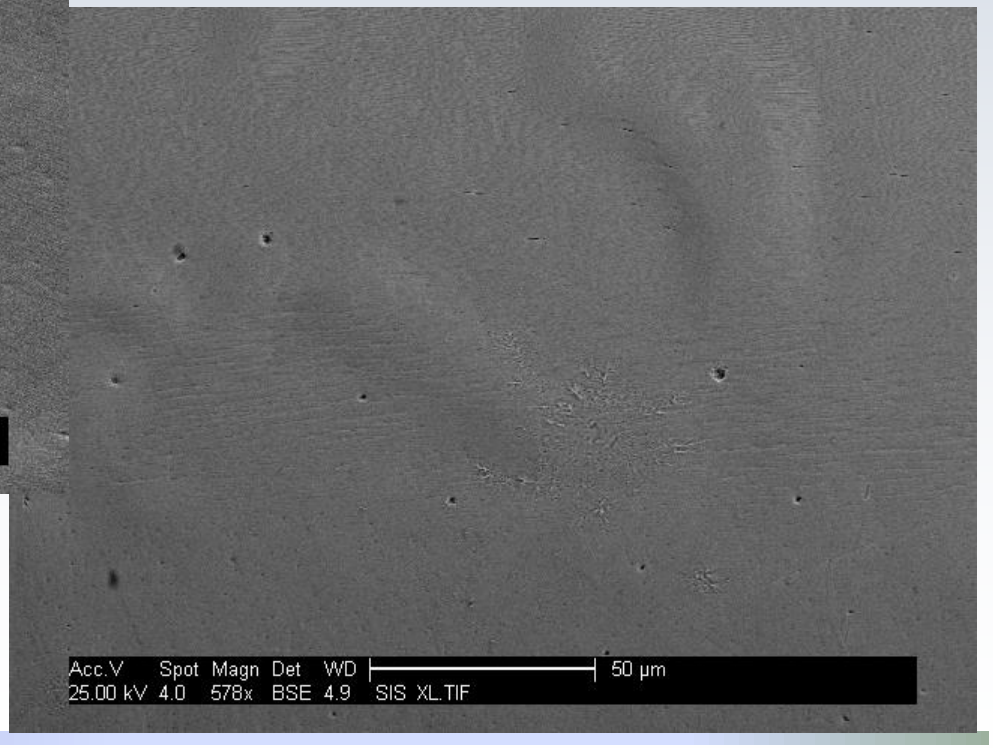




III. RF-tested disk#23

- Surface deformation – “skeleton” observed better when a sample is tilted

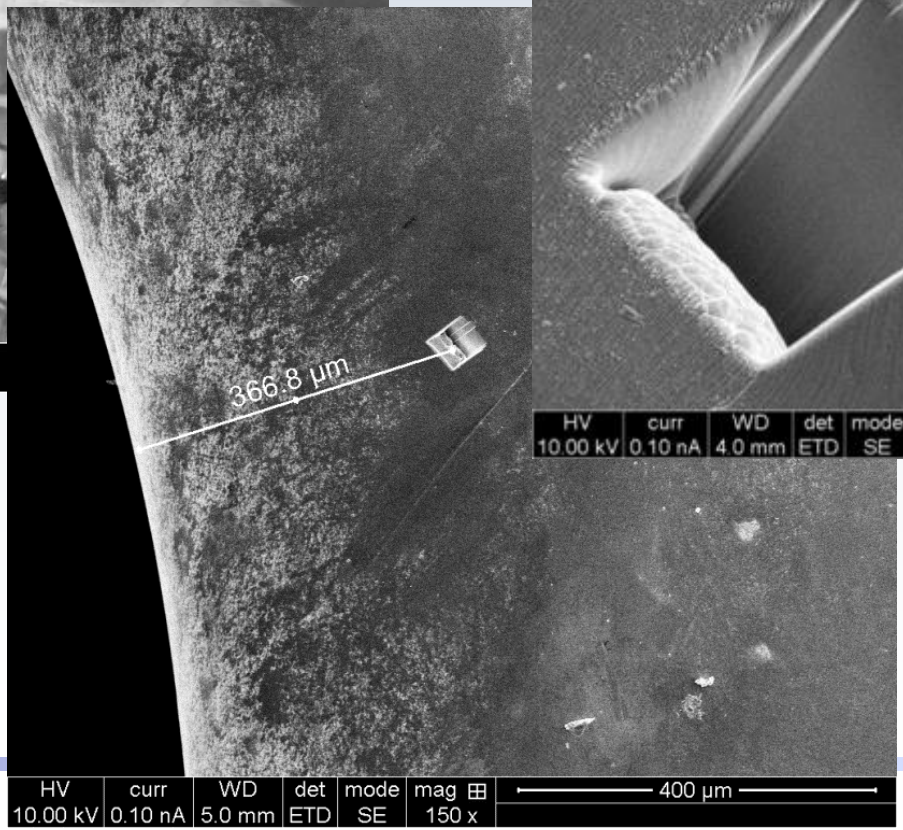


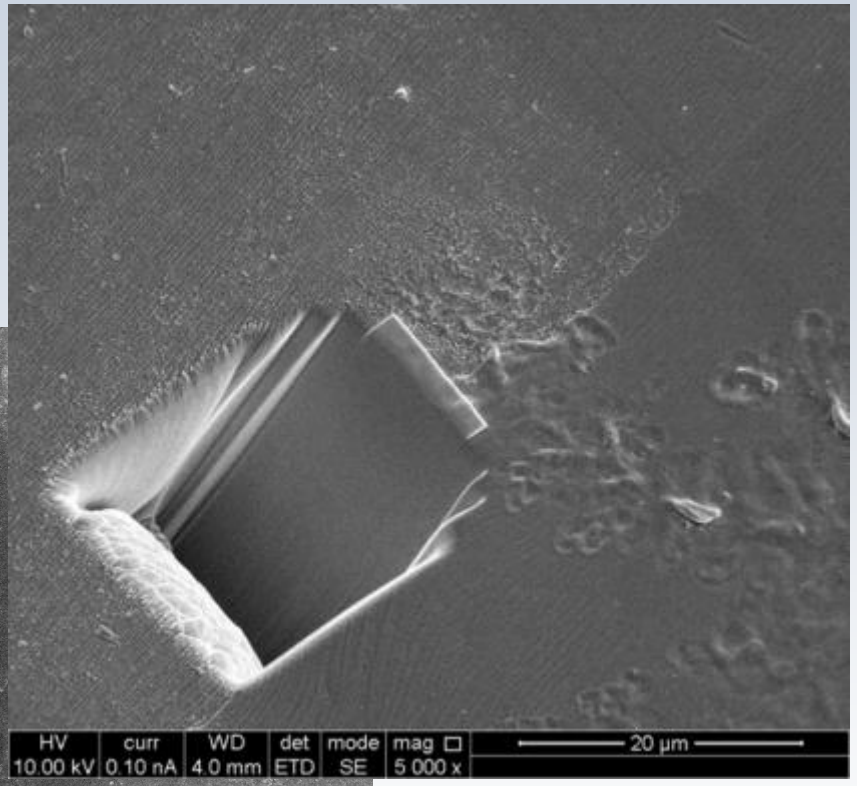


III. RF-tested disk#23

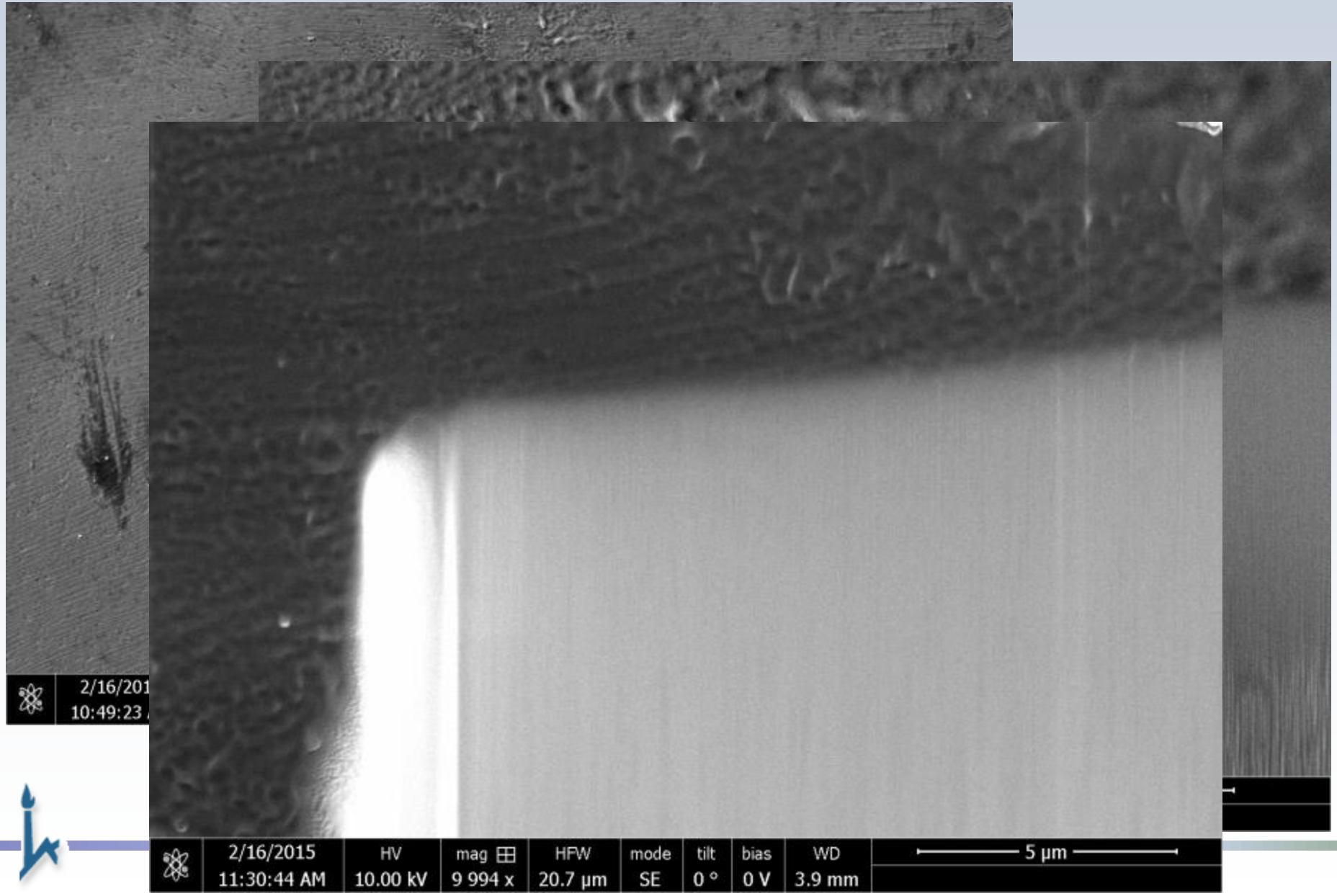
- ✓ **Defects containing W and C - machining of soft Cu**
- ✓ **Surface oxidation**
- ✓ **Surface organic contamination always presents**

III. RF-tested disk#23 – Cross-sectioning
FIB milling across the “new features and GBs

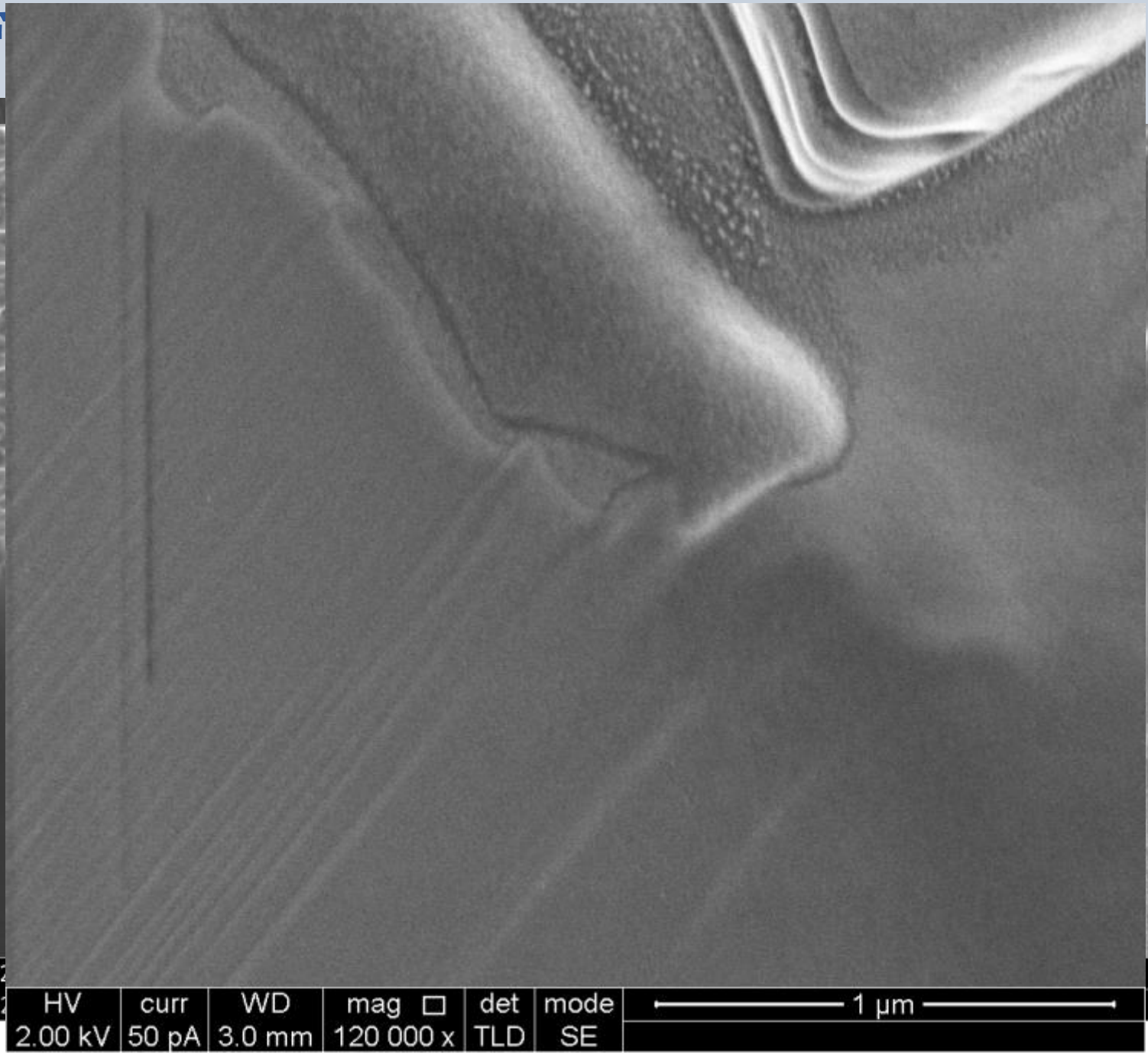




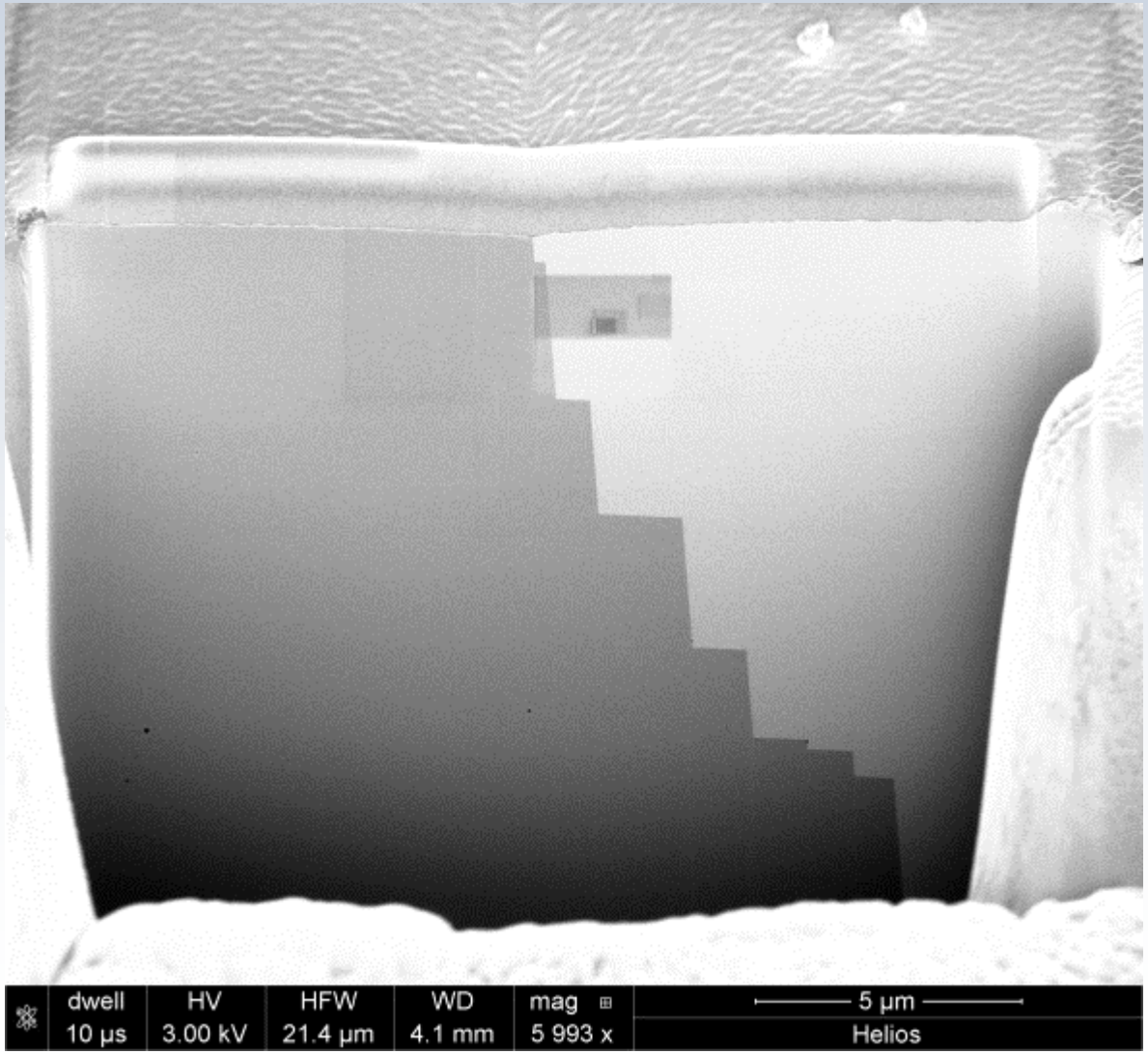
III. FIB Cross-Sectioned ROI1



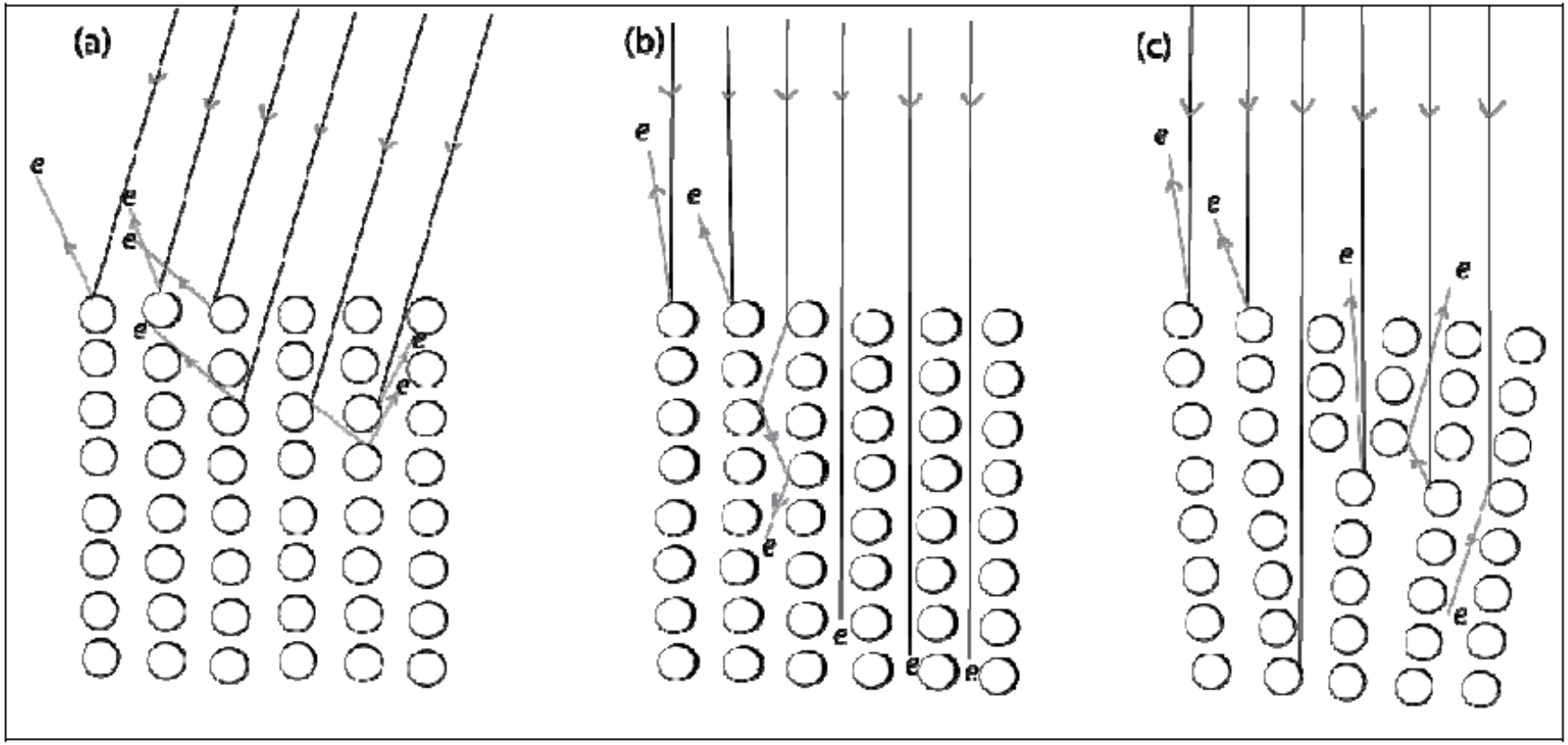
III. F



III. FIB Cross-sectioning +
+ Electron Channeling Contrast Imaging (ECCI)



Electron Channeling Contrast Imaging (ECCI)



“Closed” channel

“Open” Channel

Edge dislocation “closes” the open channel

The reported ECCI of dislocation

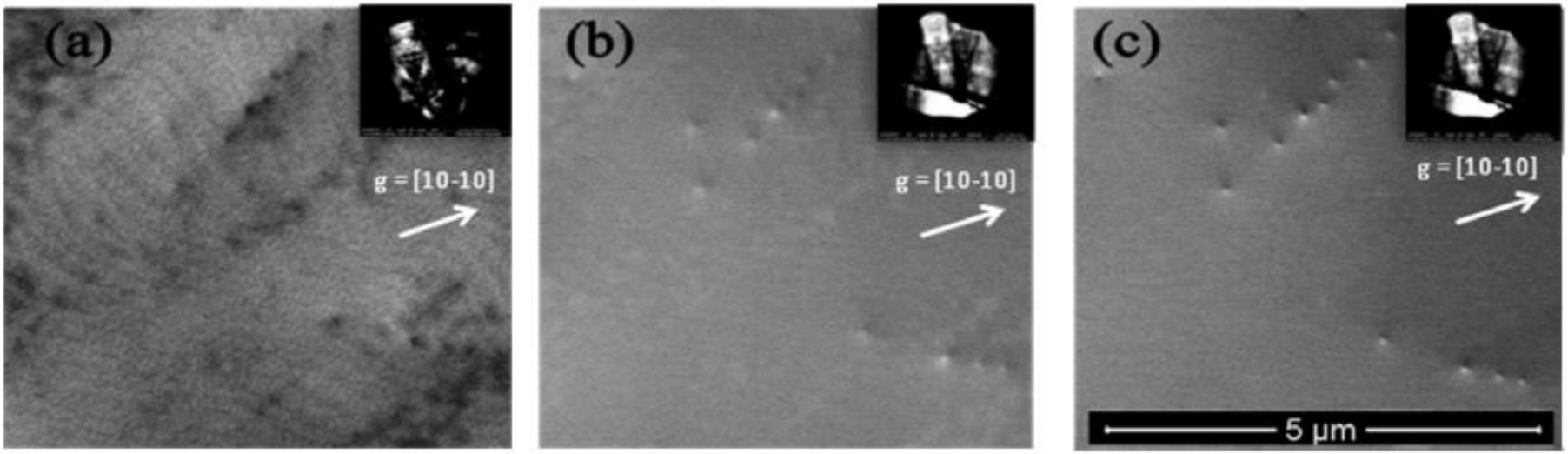


Fig. 3 ECCI micrographs showing diffraction contrast of screw dislocations in GaN (0002) at (a) 5kV (b) 10kV (c) 20kV.

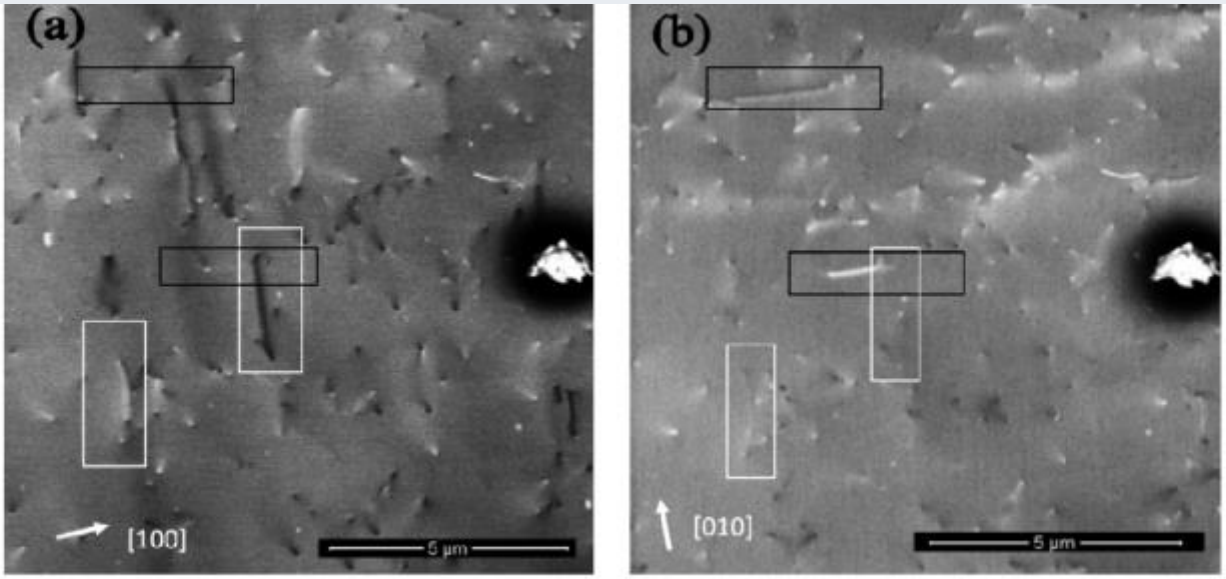
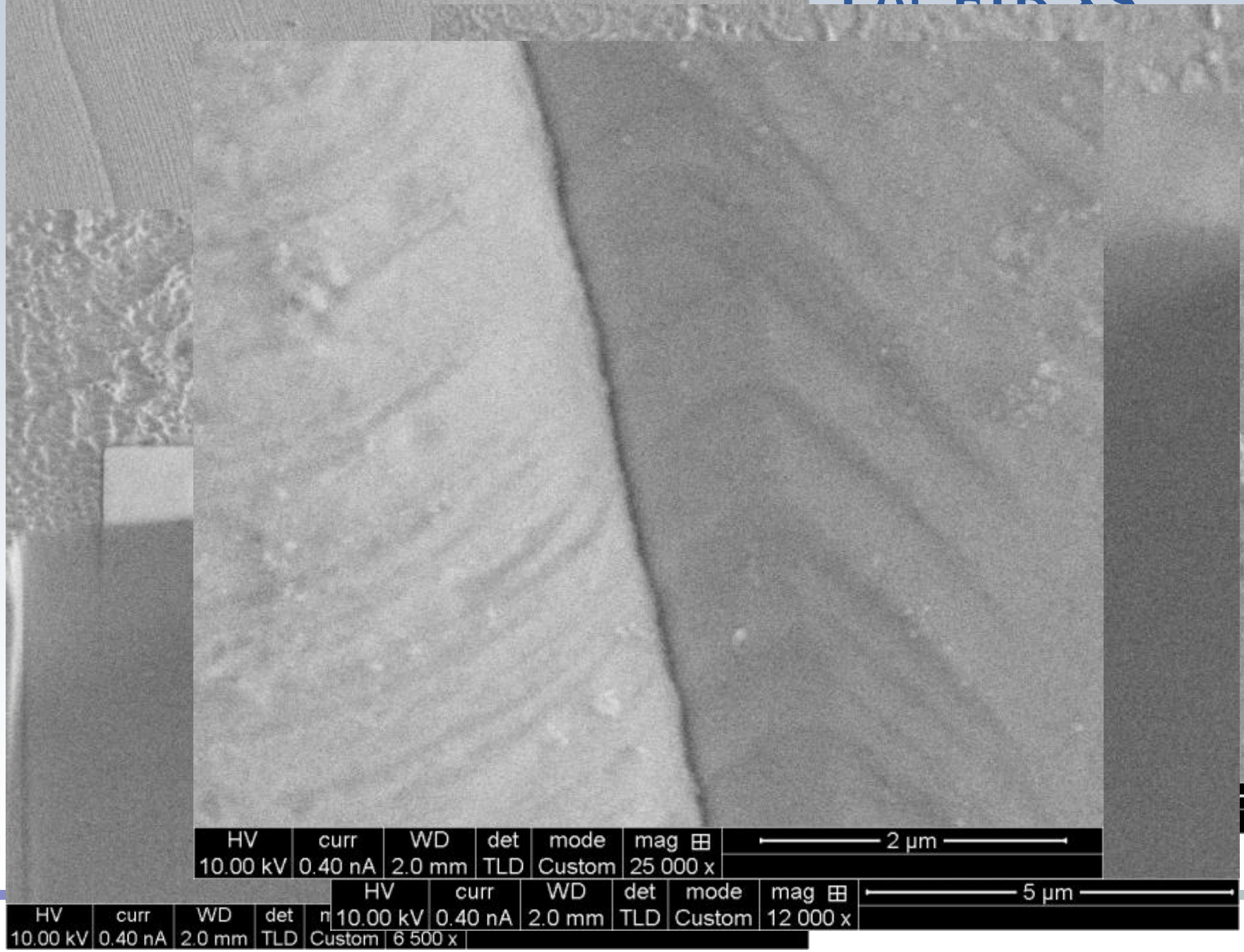


Fig. 6: ECCI micrographs of $SrTiO_3$ (001) recorded in a backscatter geometry with (a) **g** = 100 and (b) **g** = 010. White rectangles denote dislocation lines visible in (a) and invisible in (b). Black rectangles denote dislocation lines visible in (b) and invisible in (a).

CL of FIB XS


HV curr WD det mode mag
10.00 kV 0.40 nA 2.0 mm TLD Custom 25 000 x
2 µm
HV curr WD det mode mag
10.00 kV 0.40 nA 2.0 mm TLD Custom 12 000 x
5 µm
HV curr WD det
10.00 kV 0.40 nA 2.0 mm TLD
Custom 6 500 x

Questions to conclude & steps to be done:

- ✓ **Microscopic study is intrinsically non-statistical =>**

still at least a few good reference (post-production) samples must be analyzed in order to distinguish between the as-manufactured and RF-tested microstructures.

- ✓ **Can we identify the pre-BD region in RF tested samples?**

Not now, but helpful idea is under the test.

- ✓ **Is BD phenomenon related to generation/movement of dislocations?**

More structural information is needed (ECCI, EBSD, TEM to be applied)

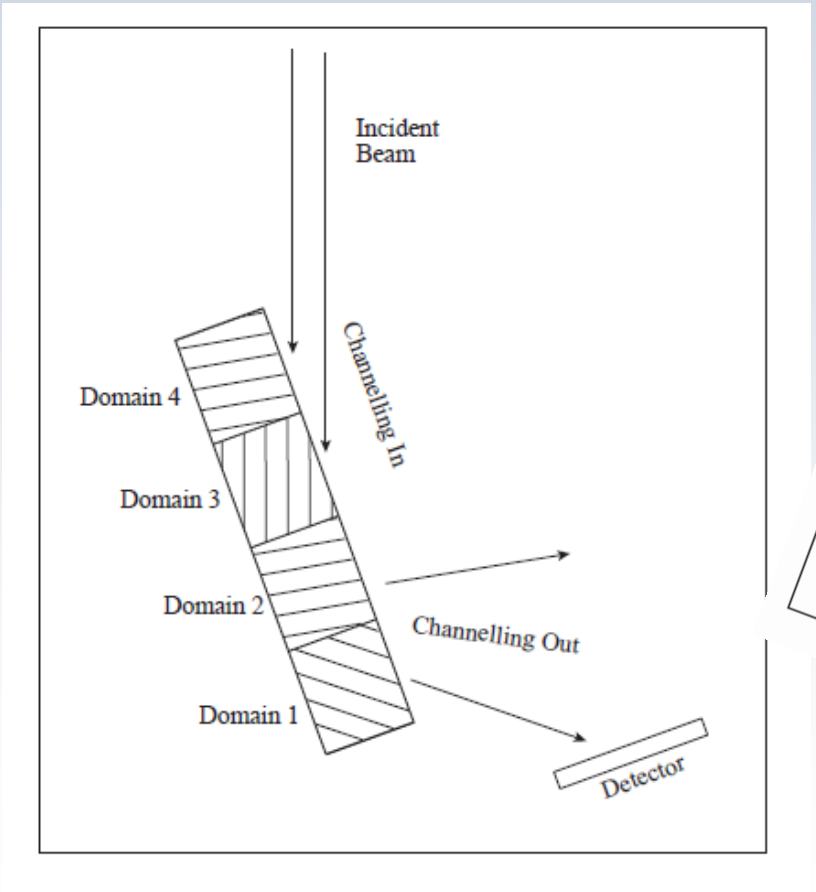
- ✓ **Is BD-phenomenon surface confined?**

- ✓ **What is the role of surface oxidation, carbon contamination, nano-scale relief ?**

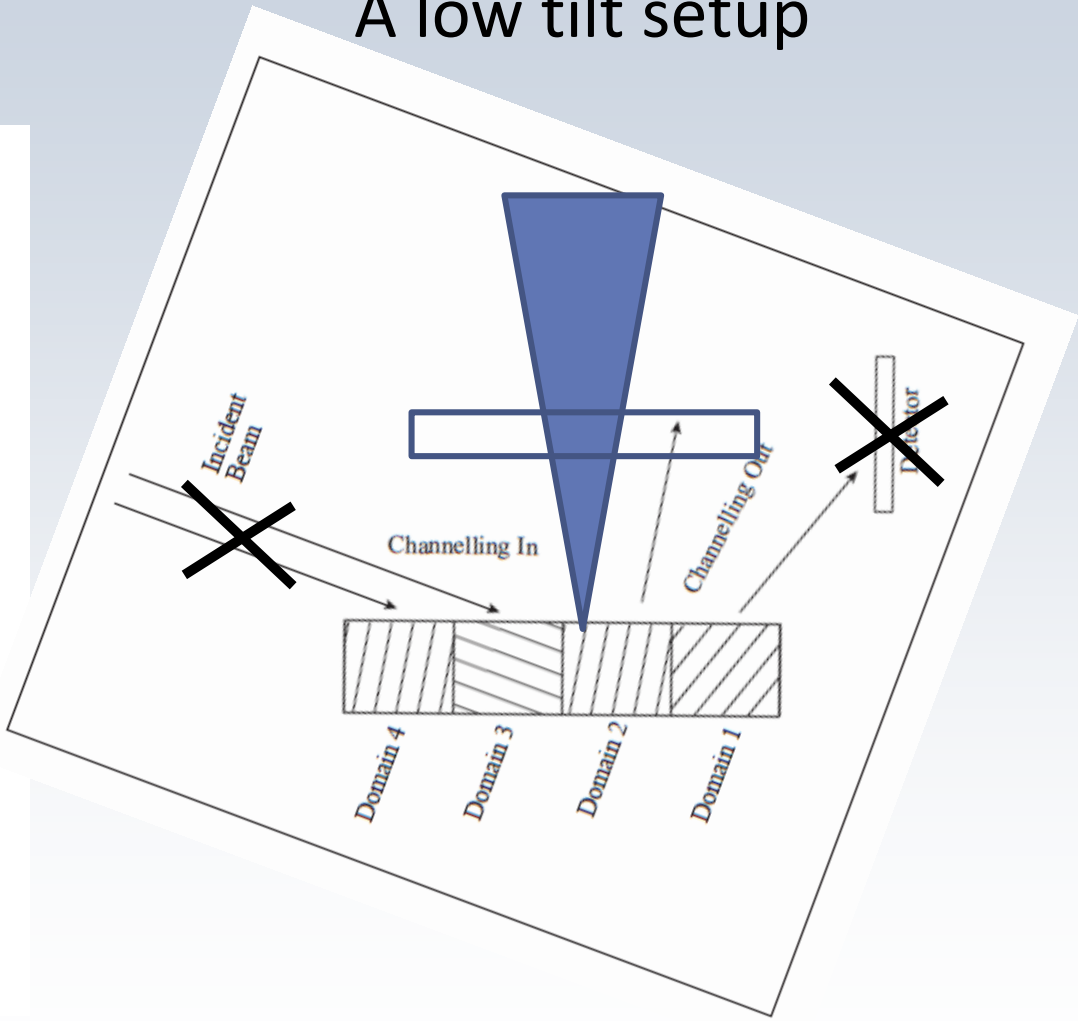
Thank you very much for your attention

Electron Channeling Contrast Imaging (ECCI)

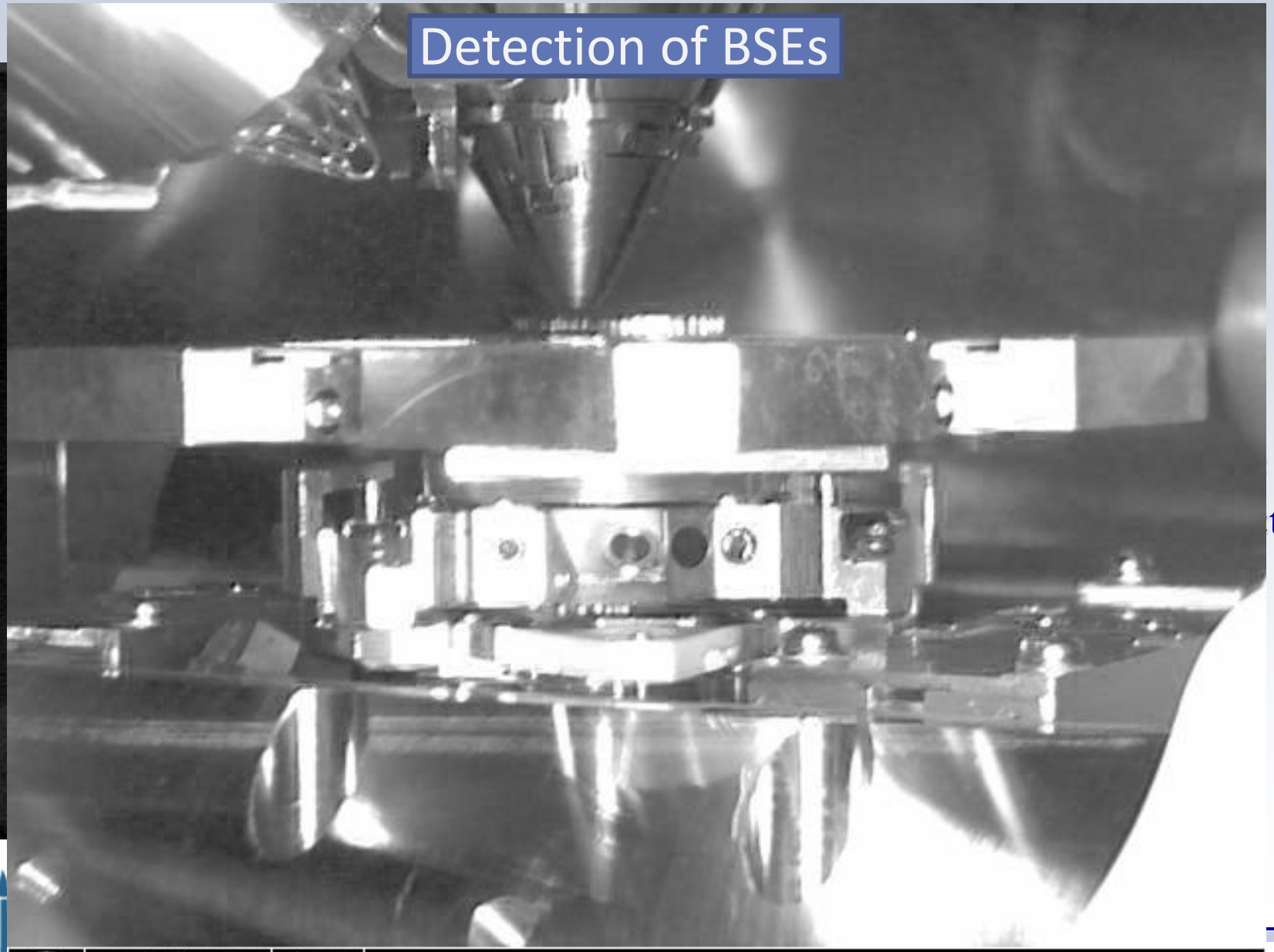
A high tilt setup



A low tilt setup



Detection of BSEs



BSE

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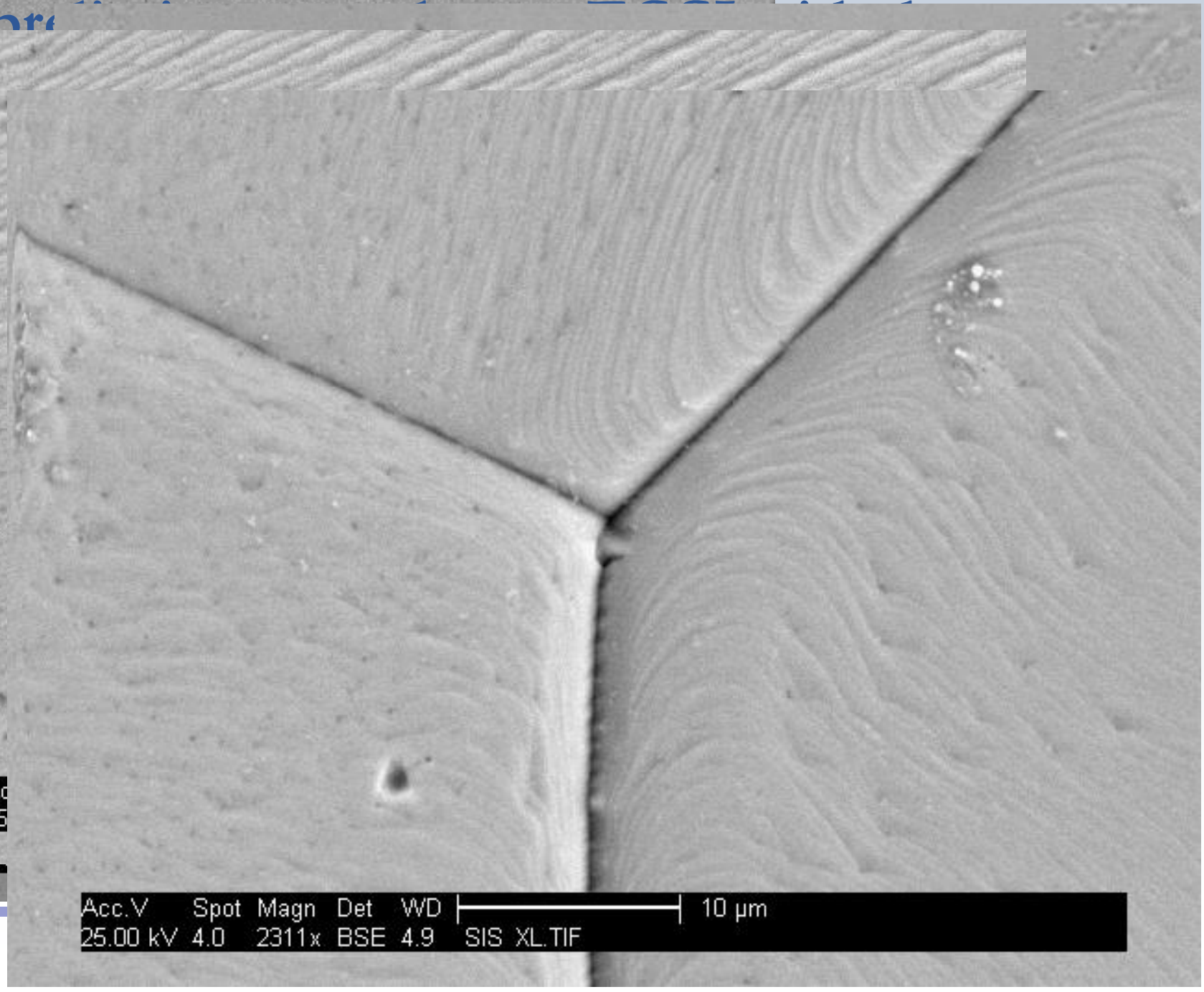

Our preliminary results on ECCI of FIB XS with BSE mode of TLD