The Harvey M. Krueger Family Center for Nanoscience and Nanotechnology

האוניברסיטה העברית בירושלים
THE HEBREW UNIVERSITY OF JERUSALEM

Constraints on BD modelling - post mortem and in situ experiments

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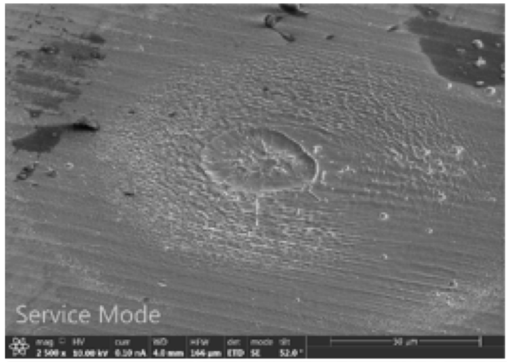
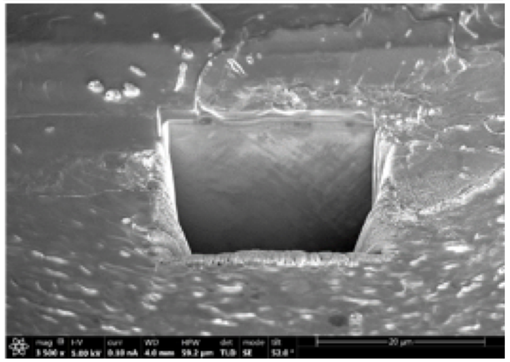
PAZY
EXCELLING IN SCIENCE

BD as a critical phenomena

High field → critical transition → nucleation → localized emission → Cu plasma → BD

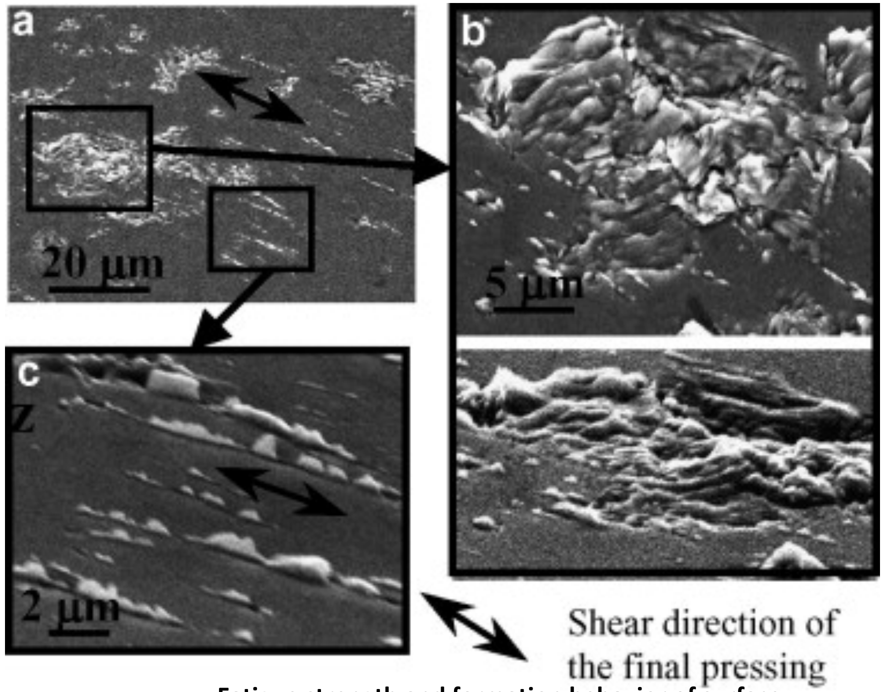
- We describe early stage BD nucleation as a critical transition in mobile dislocation population.
- Minimal model which does have:
  - Inherent sources and sinks for mobile dislocations.
  - Self interaction of dislocations.
- Observational expectations
  - No observable pre-nucleation surface structures
  - Extrinsic as well as intrinsic conditioning.
    Intrinsic - saturation of dislocation structure





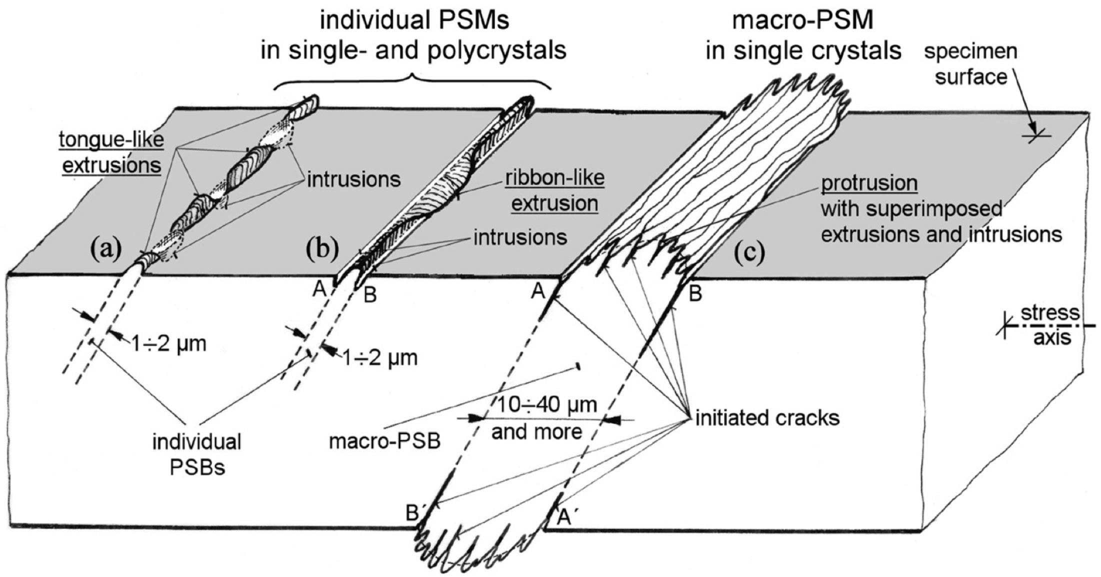
PSB -> protrusions

- Previously observed in fatigued surfaces.
- Significant sub-surface PSB leading to these surface features.
- Stochastic response at sub-yield stresses.

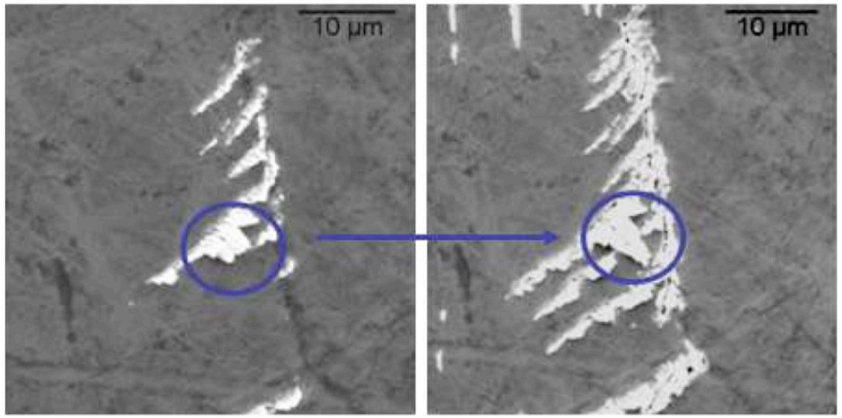


Fatigue strength and formation behavior of surface damage in ultrafine grained copper with different non-equilibrium microstructures

M. Goto et al. Int J of Fatigue.

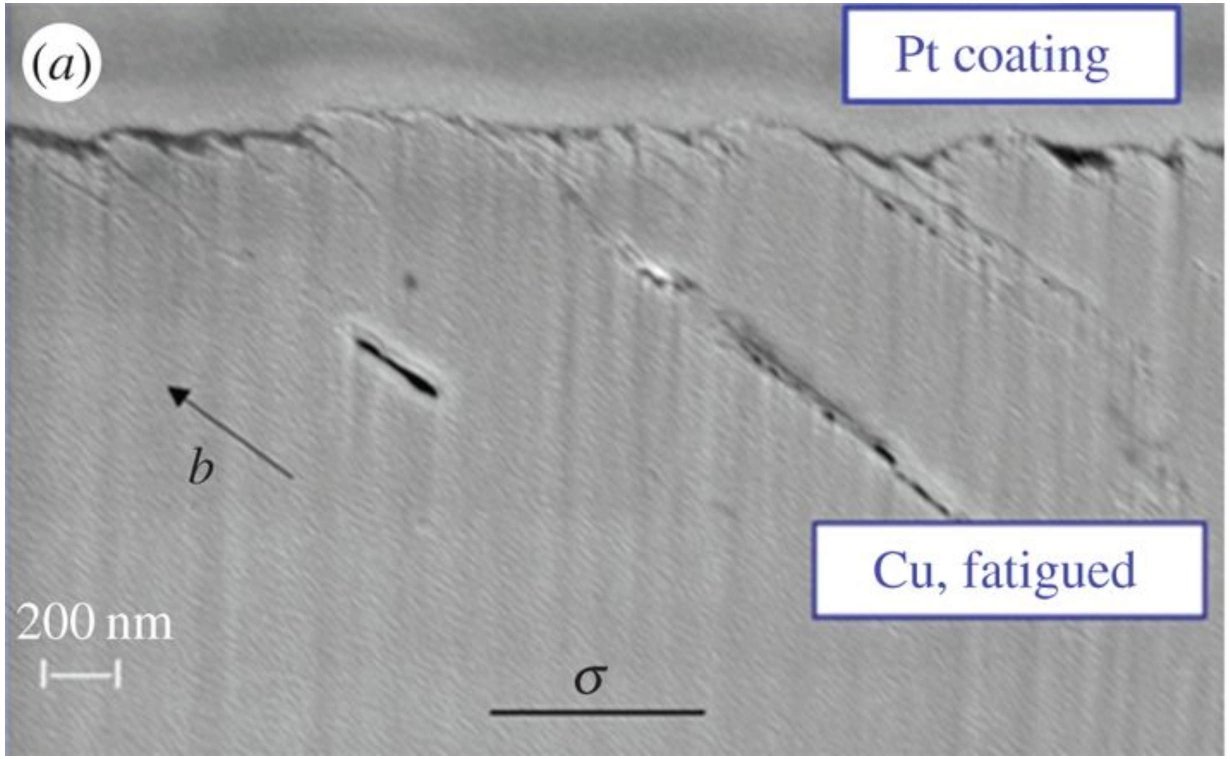


J.Man et al, Phil Mag 89 (2009) 1295

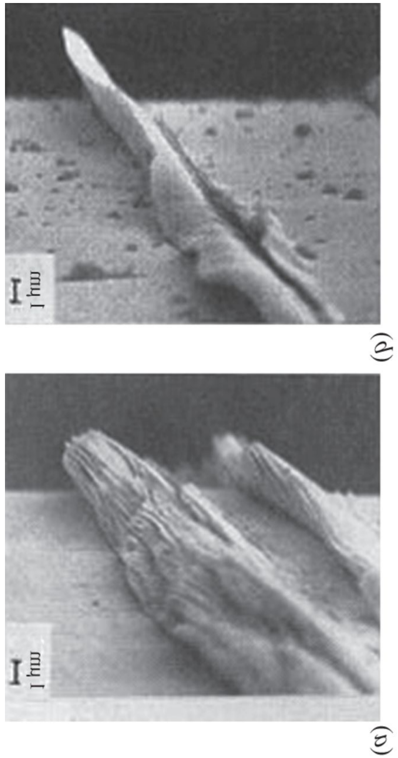


Laurent et.al. Phys Rev STAB 14 (2011) 41001

Plastic response - below and on surfaces… strongly driven (fatigue) “non related”



Polycrystal Cu - fatigued ~10^{10} times sub PSB threshold
Haël Mughrabi Phil. Trans. R. Soc. A 2015;373:20140132



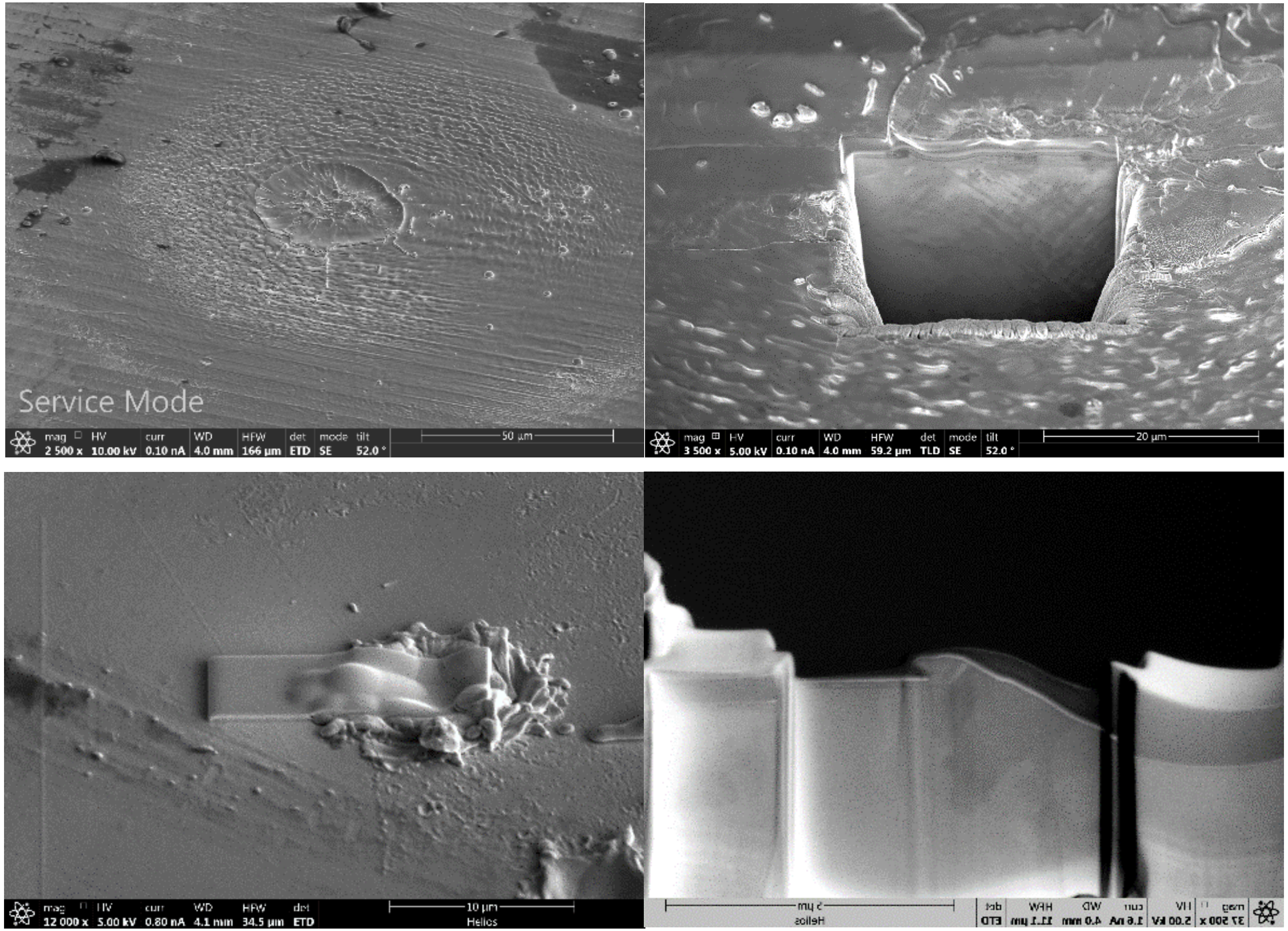
Above threshold

Previous observations…

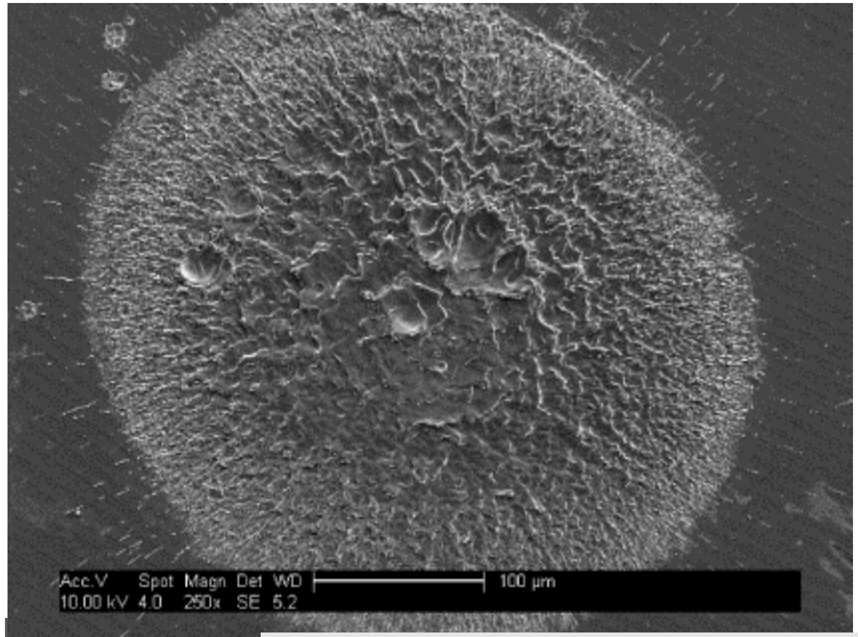
Significant plastic activity at BD sites

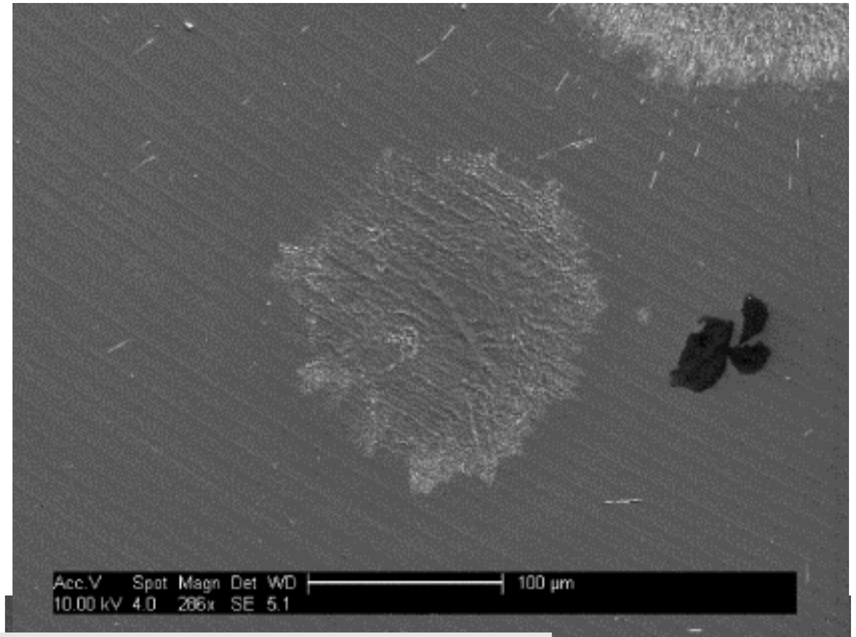
BD craters can be small (RF/DC)
Or engulfed in large pools of melted copper

But in general do not hold info on what preceded them…

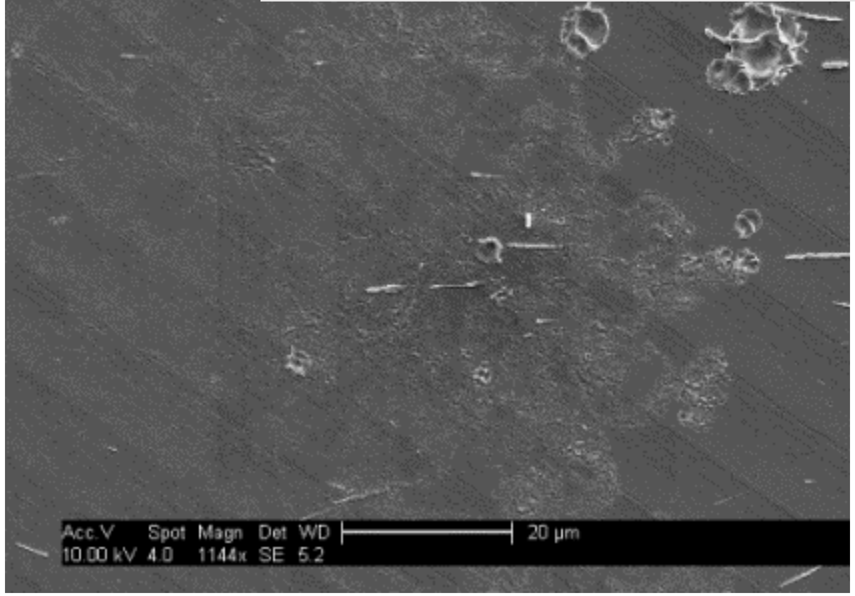


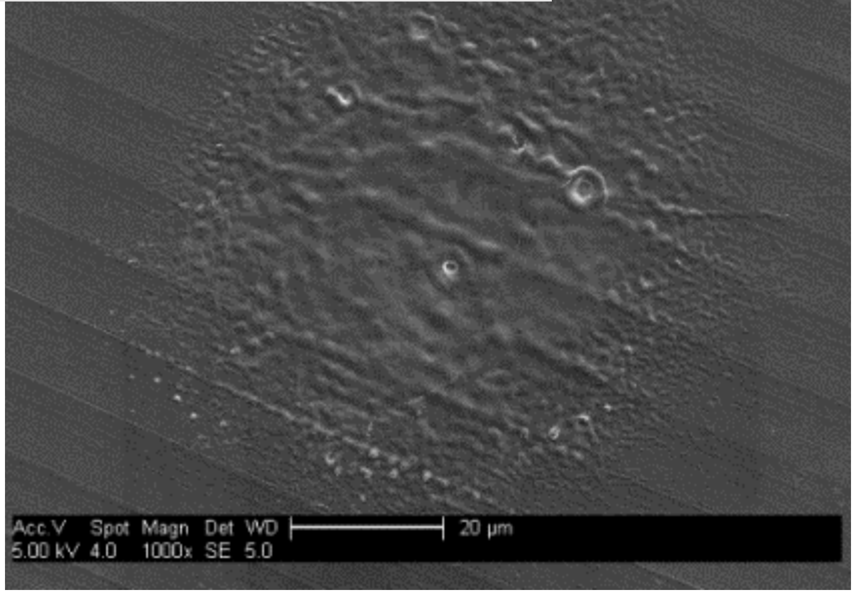
BD to sub BD





Melt formation – but no discharge pressure





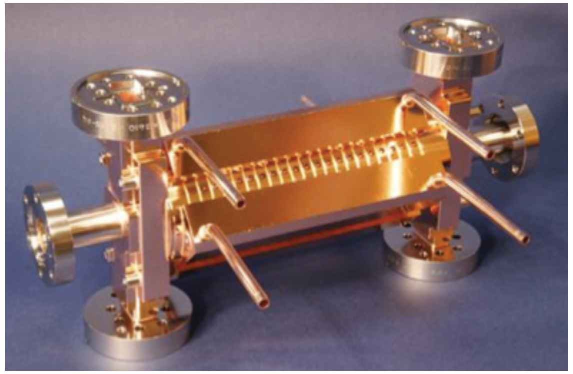
Systems and Samples

Two main types of samples

Radio Frequency (RF)

Full accelerating structures

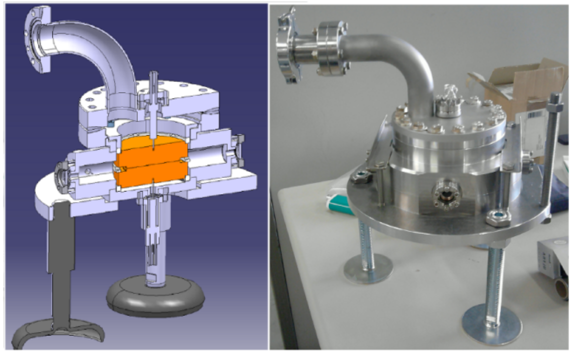
Were exposed to an RF field without an accelerated beam.

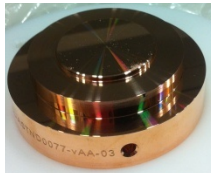


Direct-Current (DC)

Fixed gap system – Flat Anode (FA) system

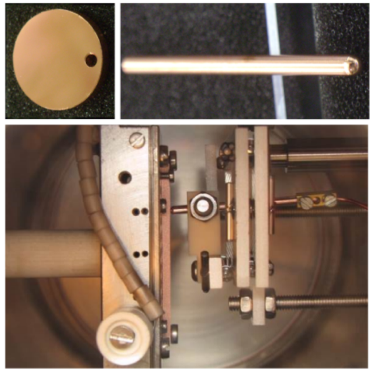
Exposed to a DC field for periods of 1μs at 1kHz.





DC spark system– Sharp Anode (SA) system

Constant DC field exposure which is generated by a localized anode tip.



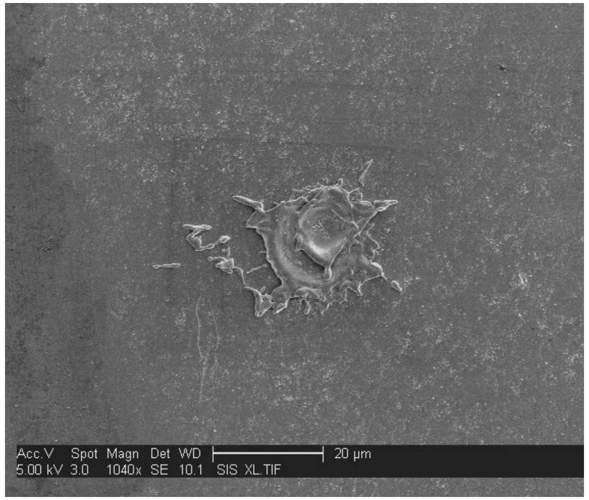
All the above systems operate under ultrahigh-vacuum (UHV) conditions. The material of all the electrodes described in this proposal is oxygen-free high thermal conductivity (OFHC) Cu, all of which were produced at CERN

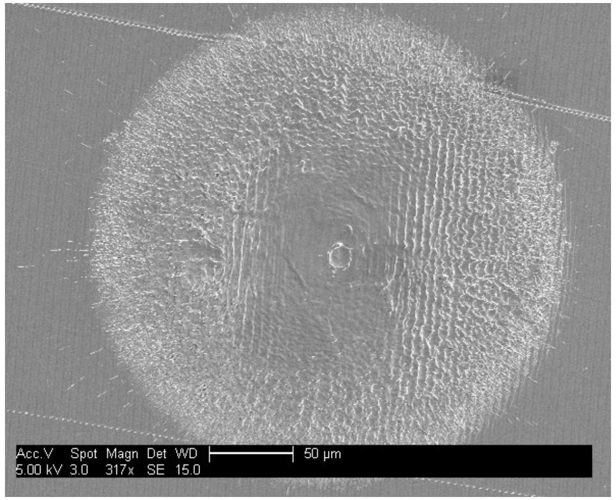
Correlation between RF and the various DC setups

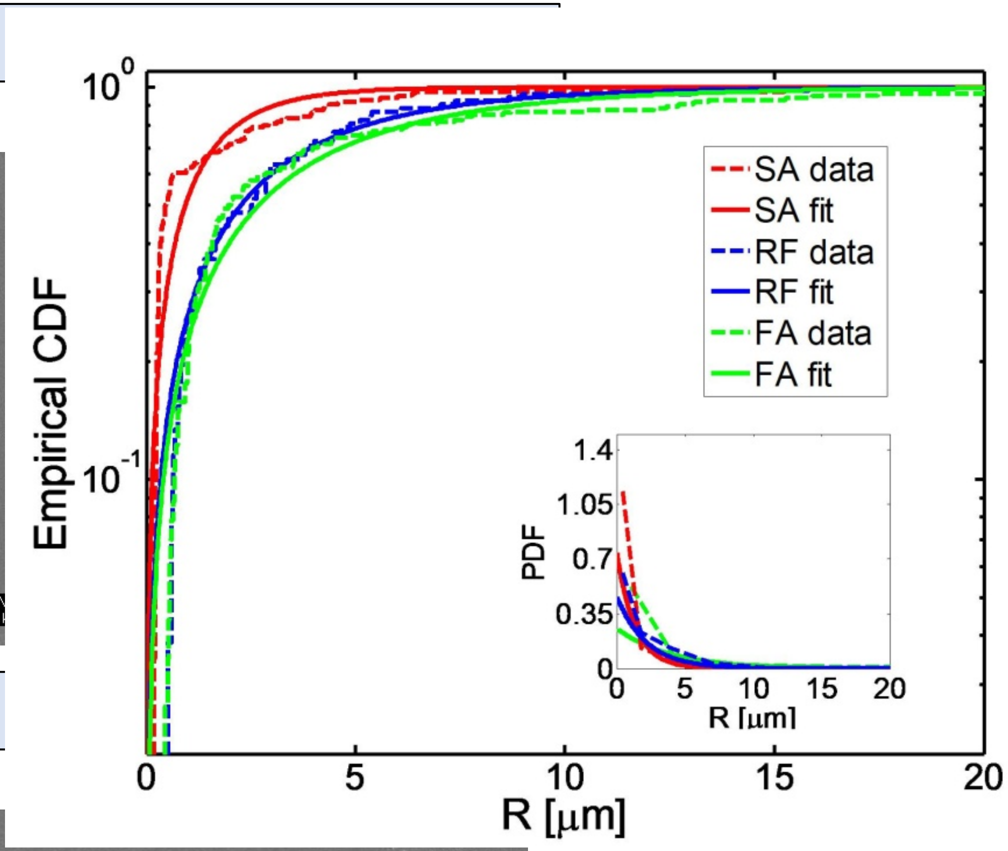
BD spots

RF

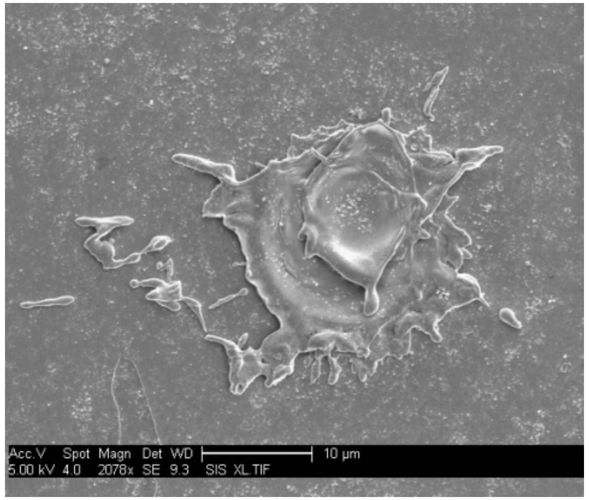
FA

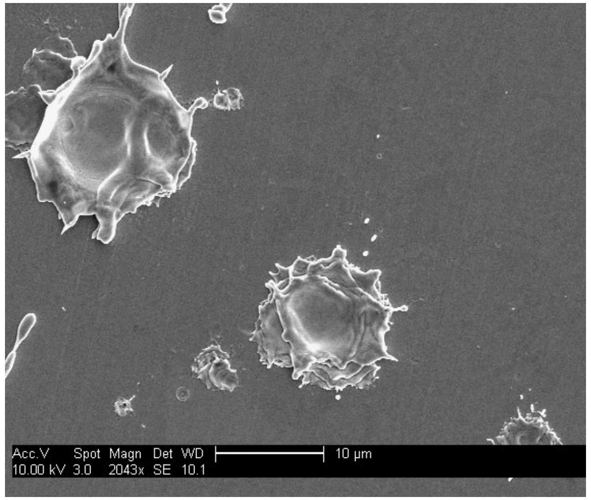


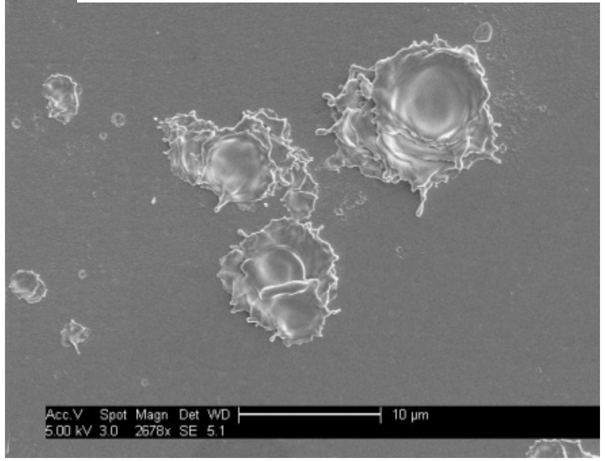




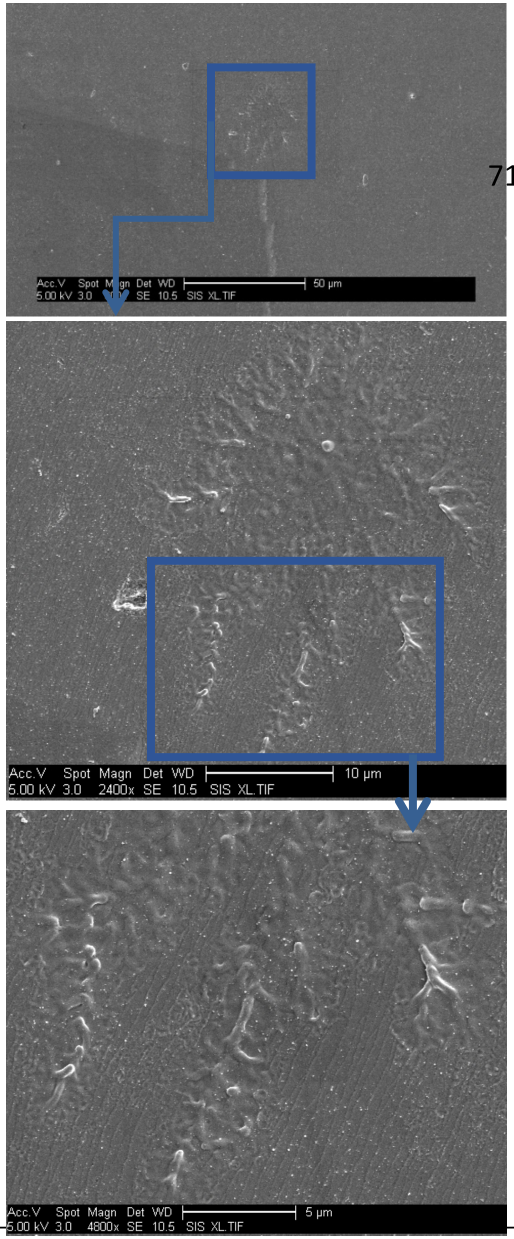
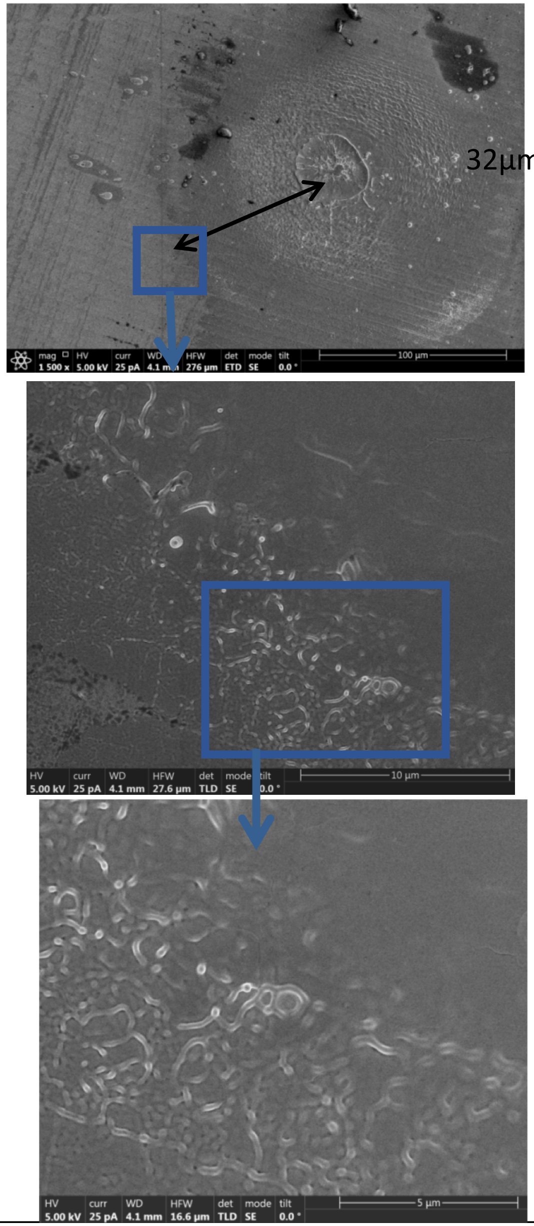
Isolated BD craters







We do consistently observe Superheated Copper far from BD

	Dendritic arms		
	RF #23	FGS #1	DC #2
Images	 71µm	 32µm	

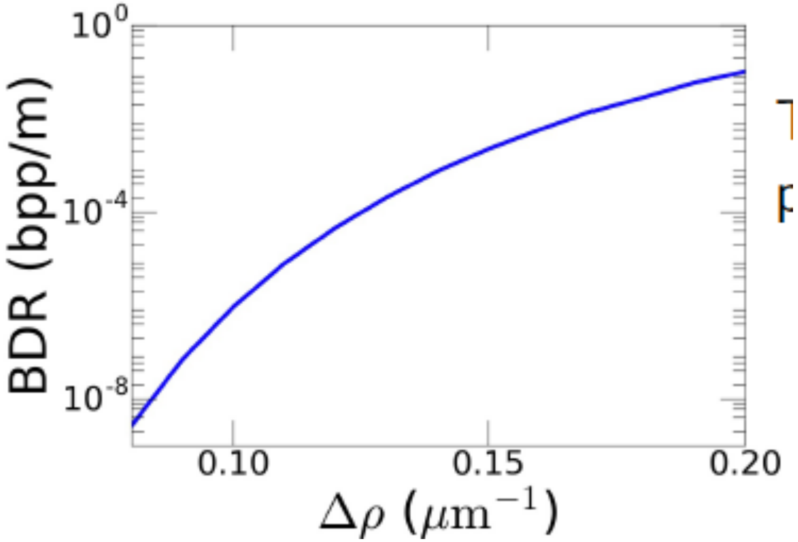
What are we looking for

- First of all – relation RF-DC ….
  - Proving DC and RF correlate well, helps a lot – availability, time constants, sample sizes.
  - And – putting constrains on model….

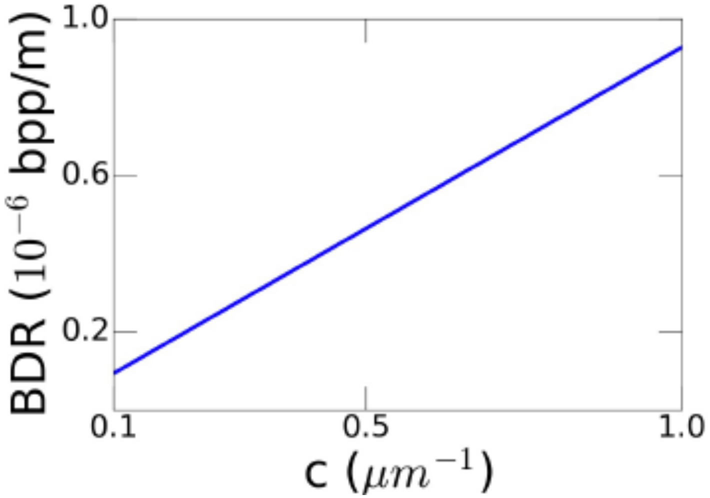
Basics of the pre-nucleation mechanism

- Stable dislocation population
- Field leads to fluctuating mobile dislocation population (which equilibrates the sessile population in a metastable state)
- Parameters limit and set the model
- But we need to look below the surface

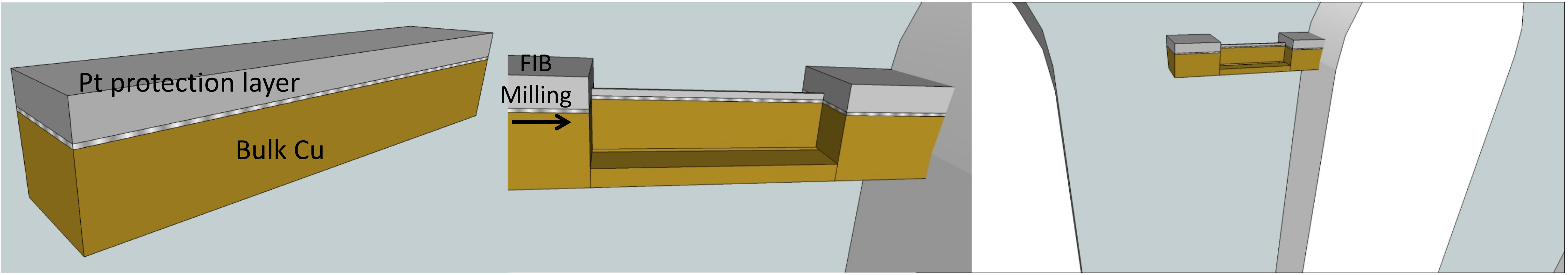
Correlation length / cell size

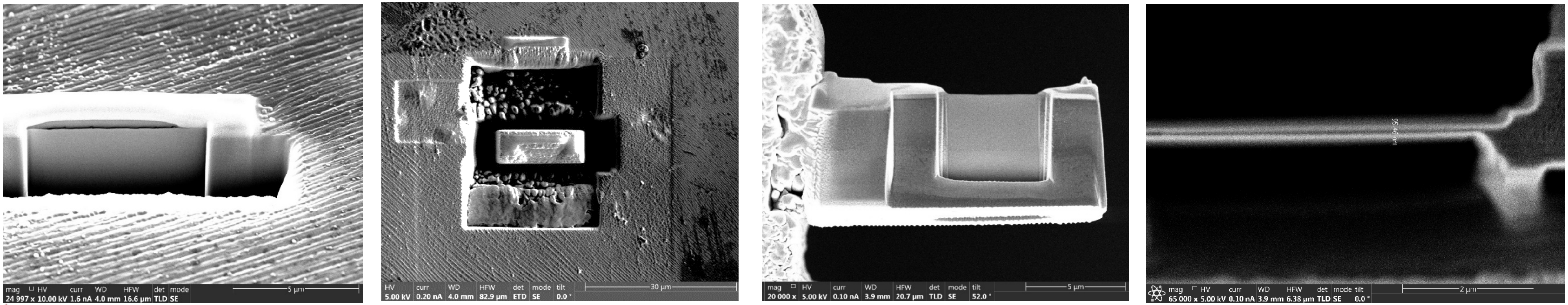


Obstacles density



Producing samples

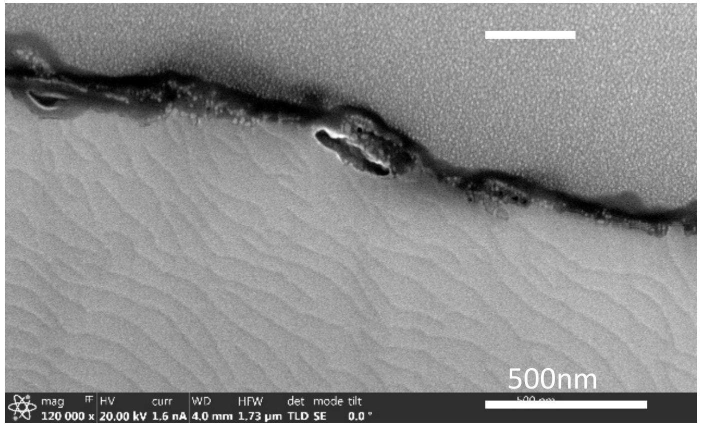




Observing dislocations

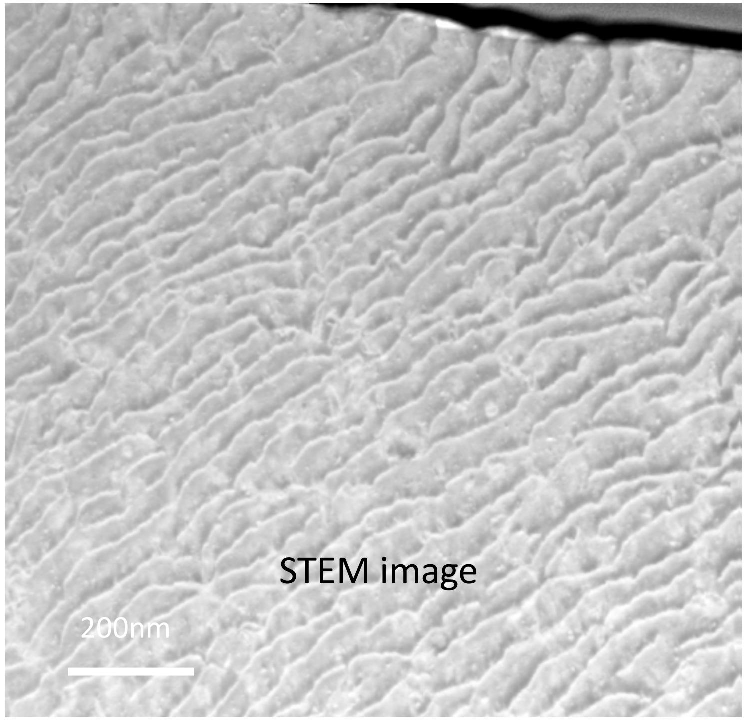
- When the cross section is made at an orientation close to {110} or {111} dislocation bands are observable and can be simply counted.

Crab Cavity below a crater

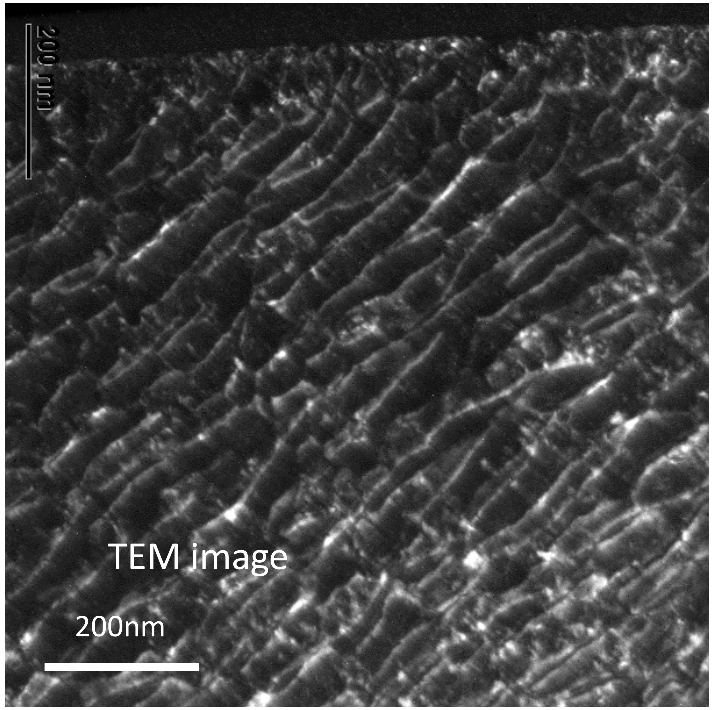


SEM image

RF system sample far from the iris

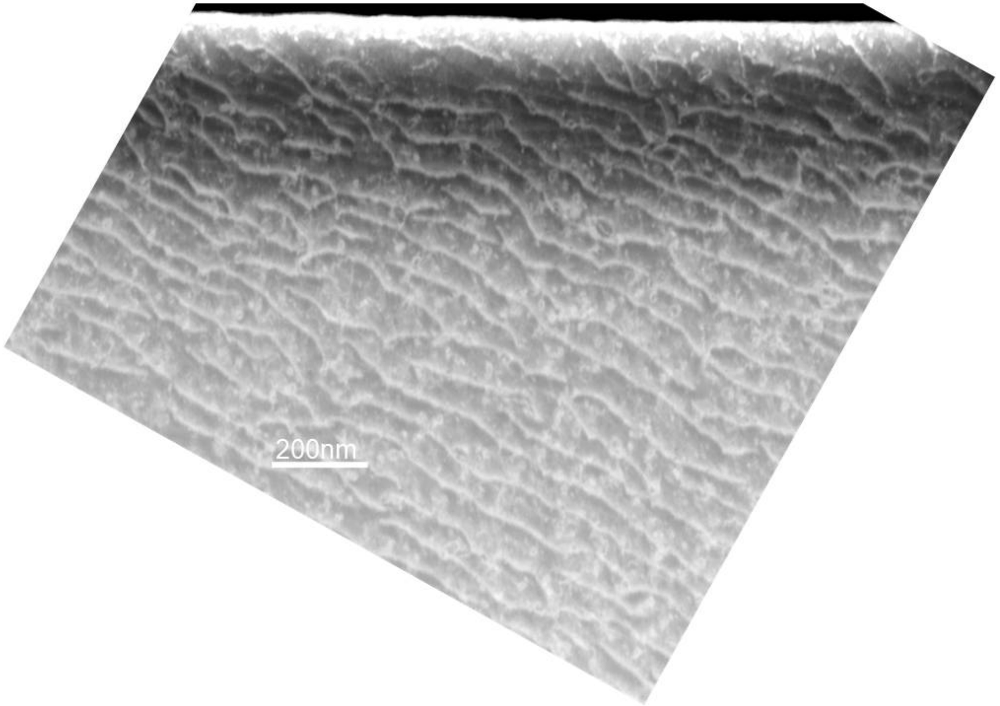


Reference sample

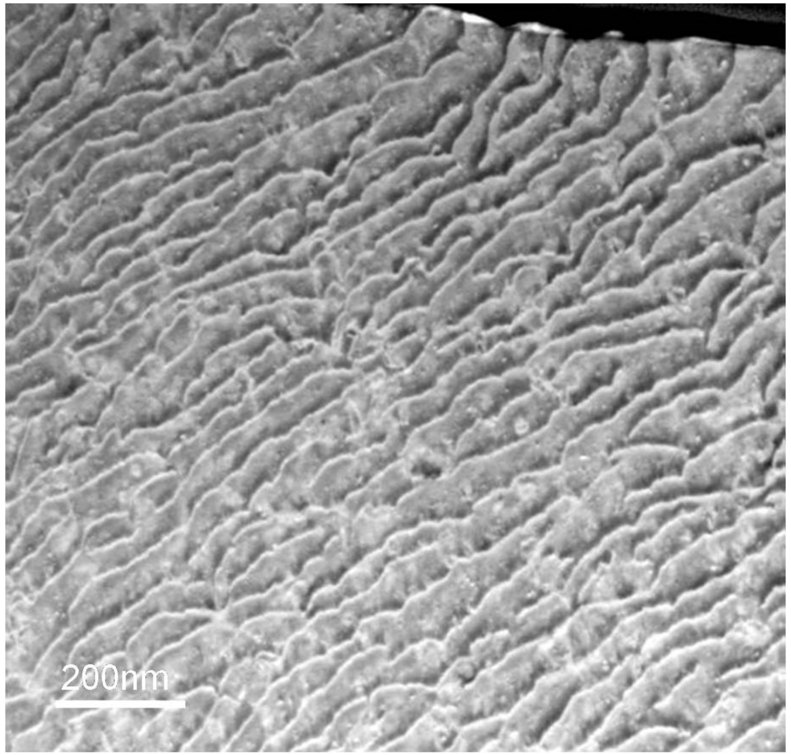


Dislocation density – pre +post exposure

Reference



RF system sample far from the iris

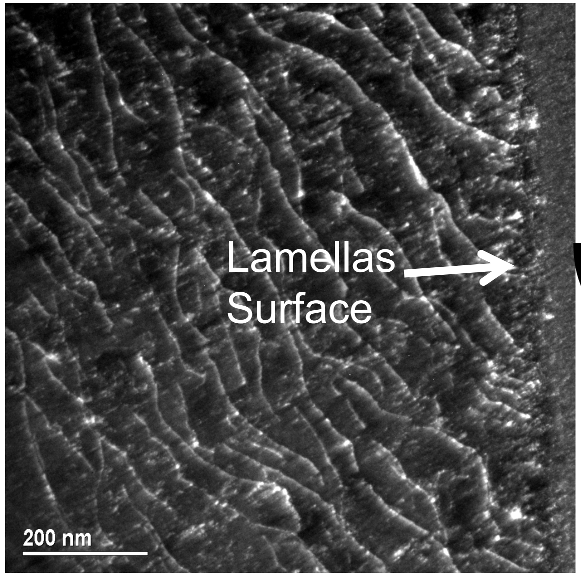


Dislocation Density

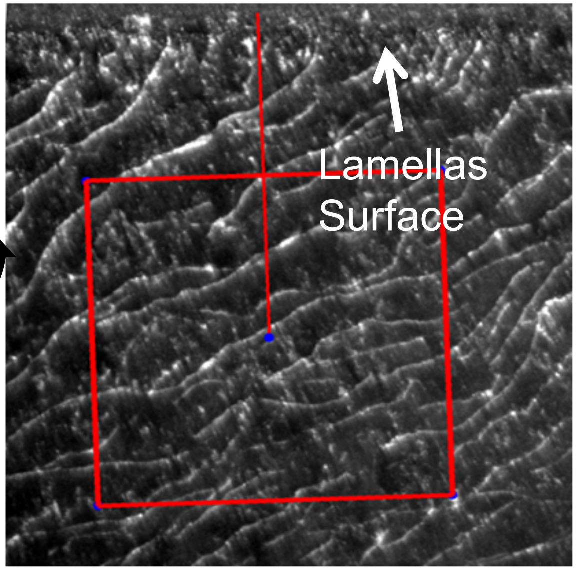
Measuring Dislocation Density Using Ham's Method*

Example: A lamella from a RF Sample

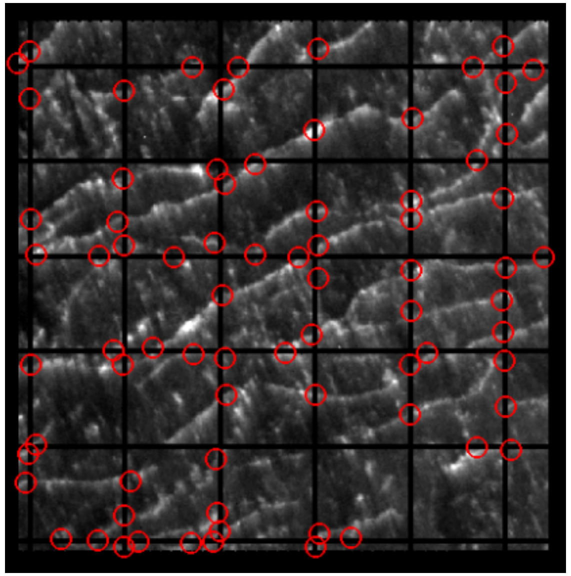
Original image



Flipped original image with the cropped region marked



Cropped region with intersections counts



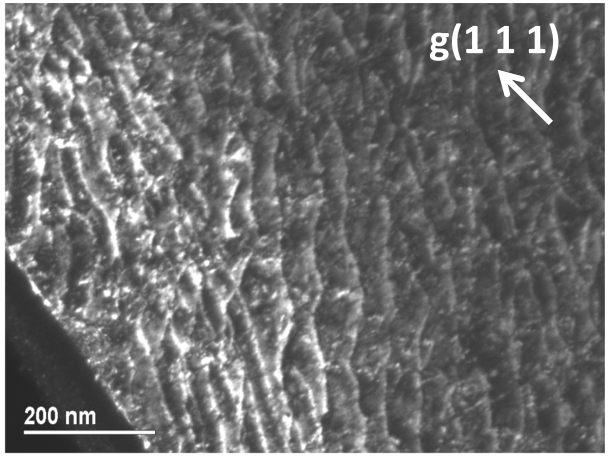
dislocation density (approximate, depends on width assumptions)

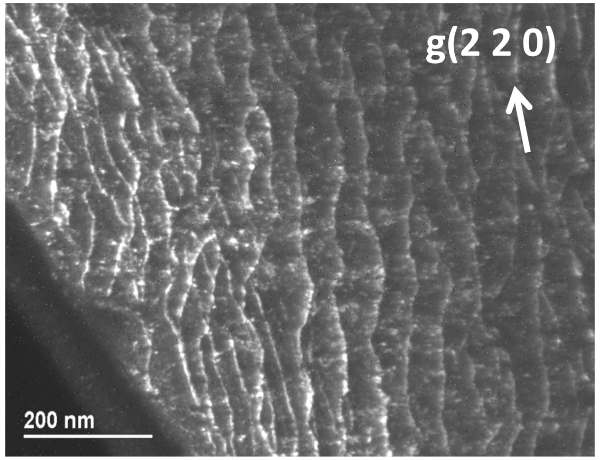
$$\rho = \frac{2N}{Lt}$$

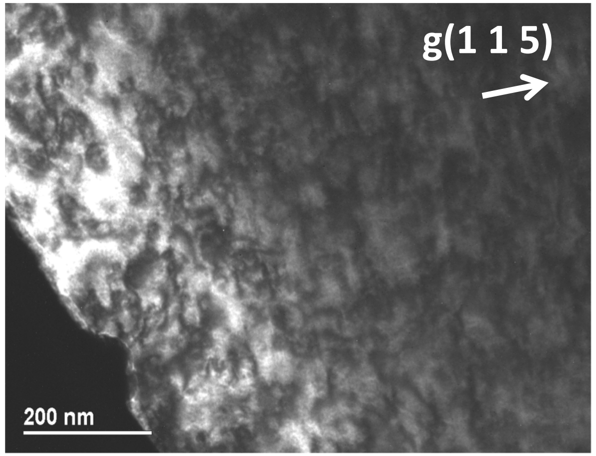
N= number of counts, L= total length of the grid lines, t = the thickness of the film

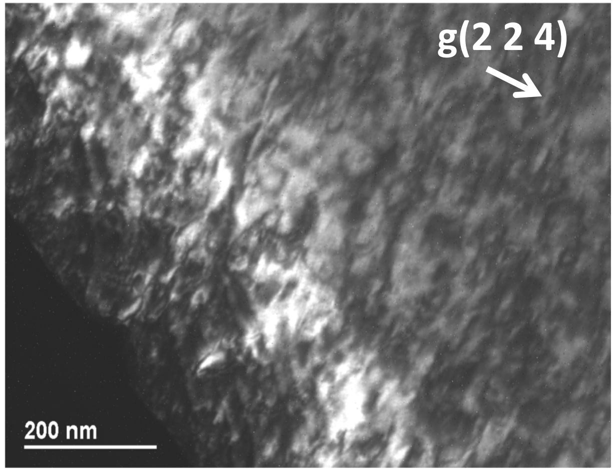
* R.K. Ham, The determination of dislocation densities in thin films, Philos. Mag. 6 (1961) 1183-1184, http://dx.doi.org/10.1080/14786436108239679

Example: Sample with no field exposure – four dark-field images of the dislocation pattern, each one with a different TB diffraction condition









➡ Dislocation contrast vanishes for TB 224 and TB 115

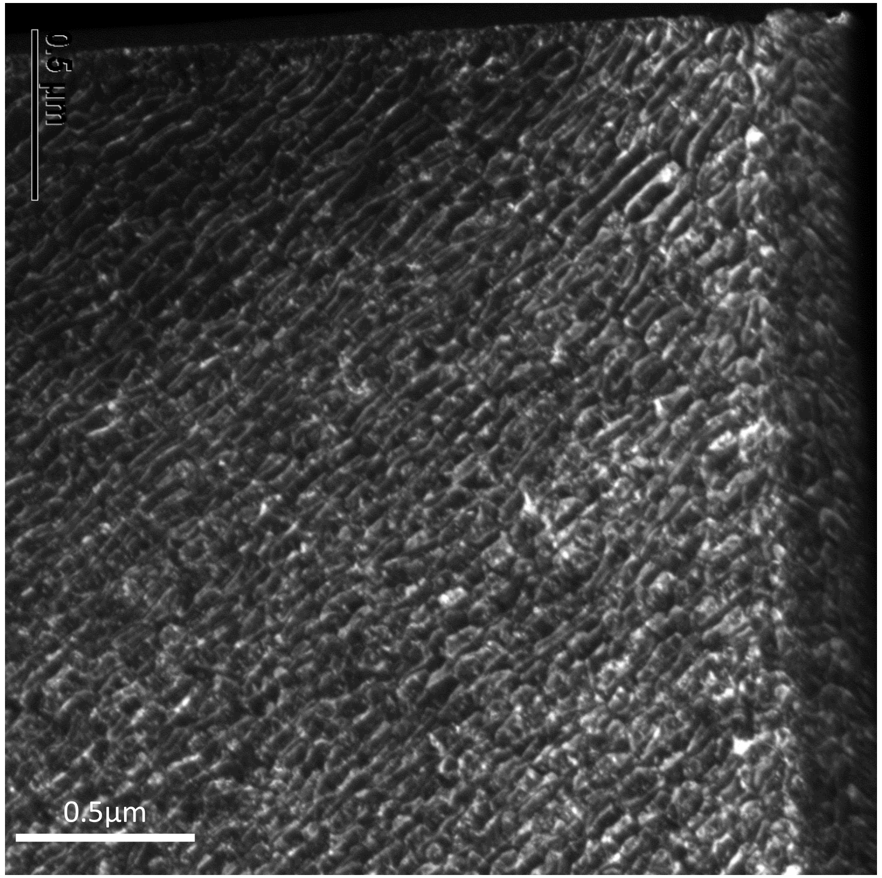
$\Rightarrow$ Burgers vector is $\vec{b} = [1\,\bar{1}\,0]$

Burgers Vectors of Two Dislocation Patterns

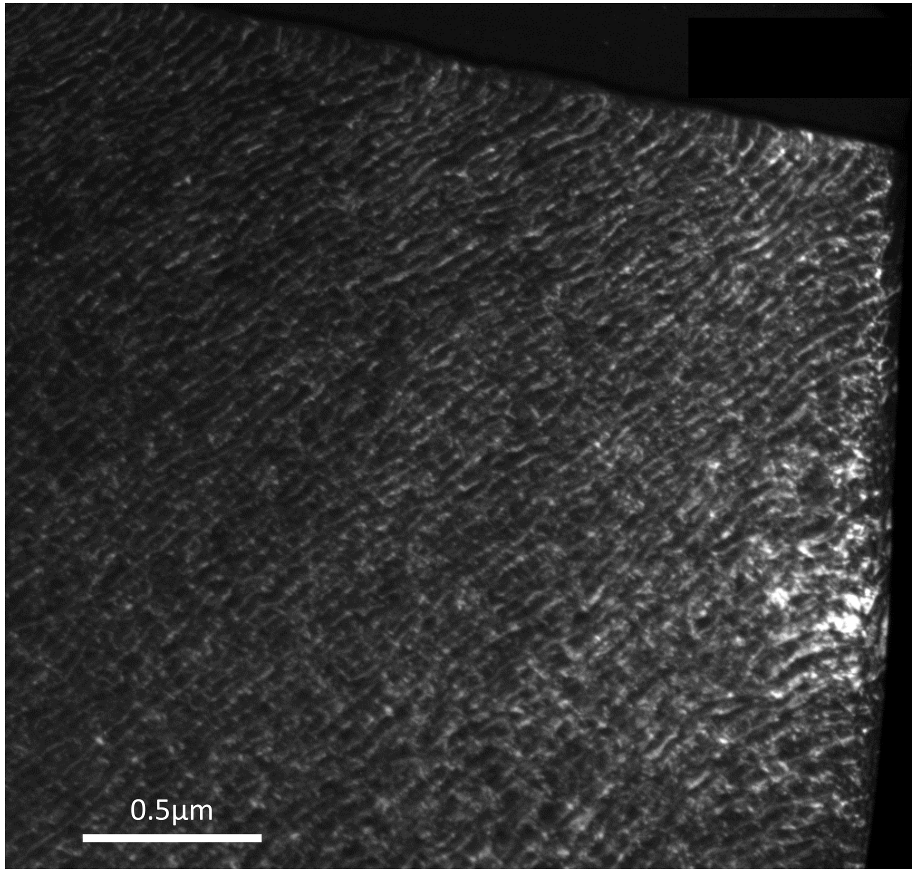
Sample Type	Field Exposure	Lamella Type	Burgers Vector
RF23	Yes	Cross section	[0 -1 1]
reference	No	Cross section	[1 -1 0]

Comparing soft single crystal Cu - pre and post field exposure (not at BD)

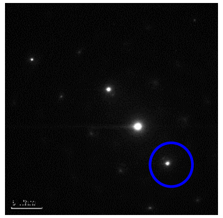
Reference Soft Cu grain 2



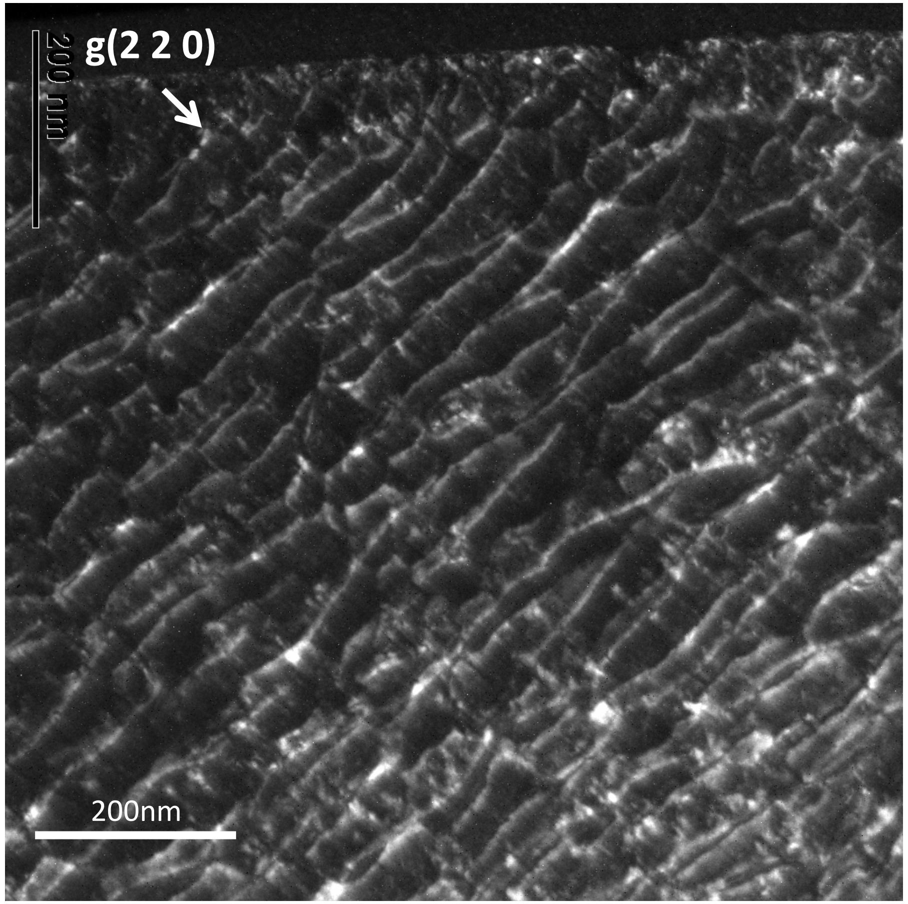
RF Sample TD24-S23 (RF23)



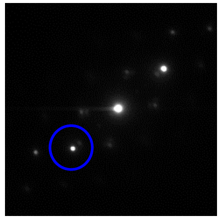
Higher density – after field exposure

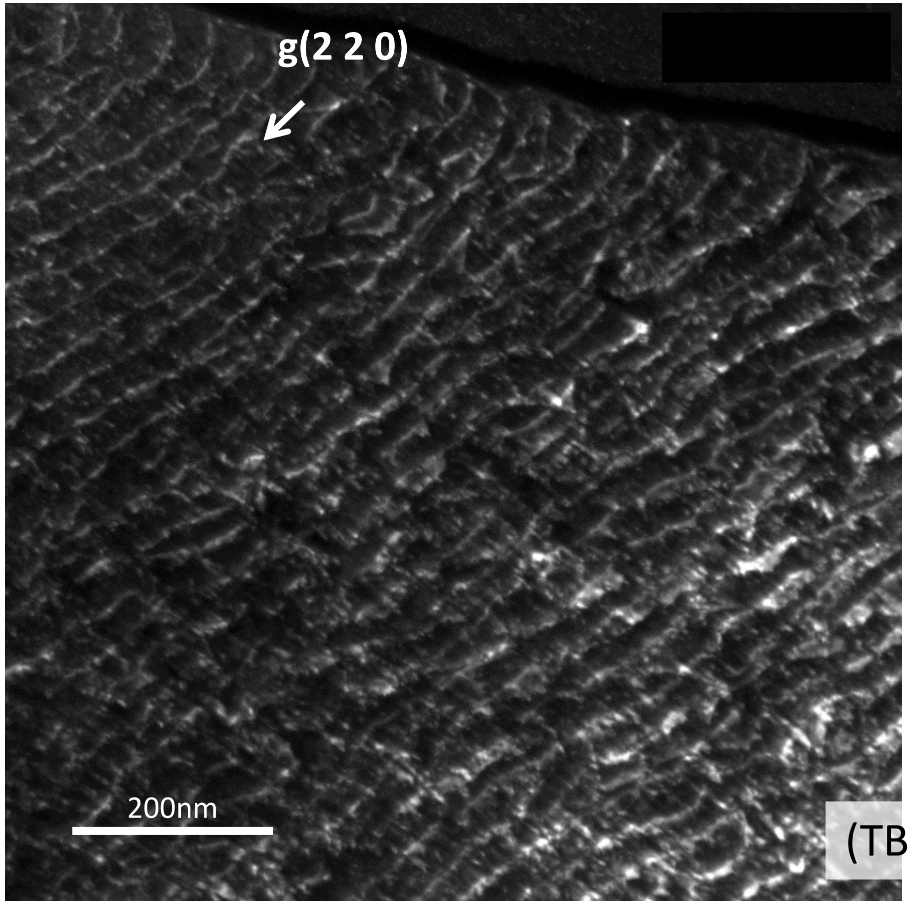


Reference



RF 23

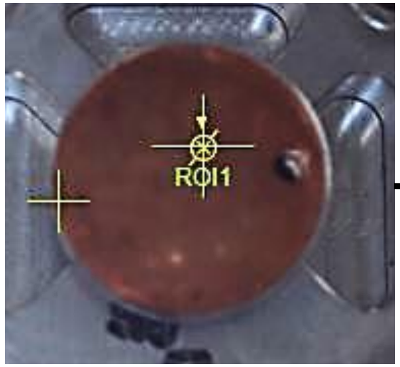


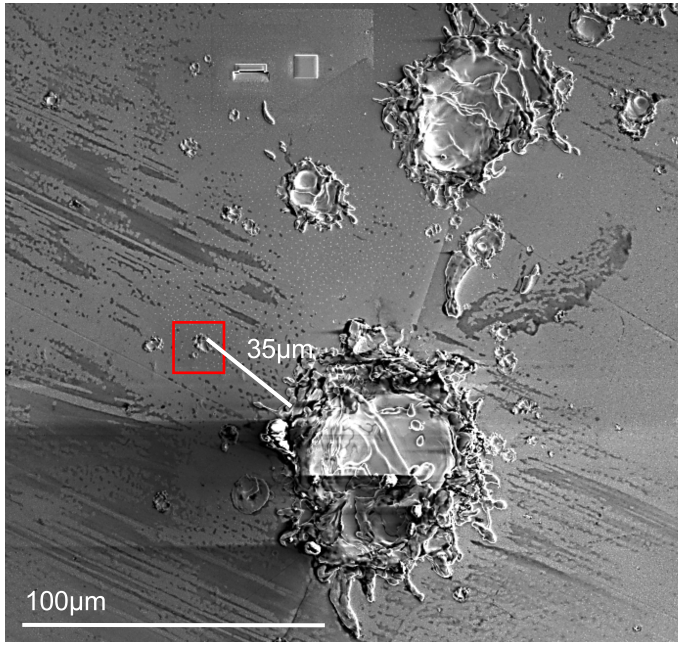


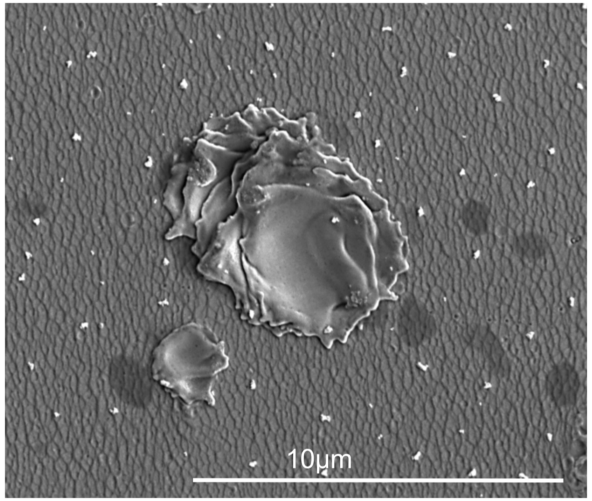
(TB 220) diffraction

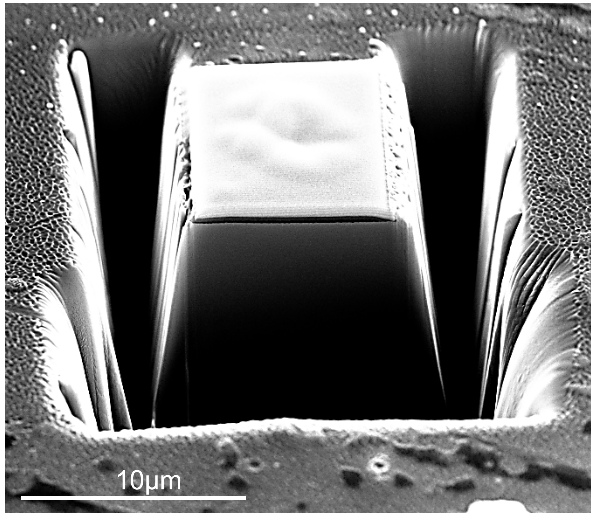
Tomography at a Breakdown Site

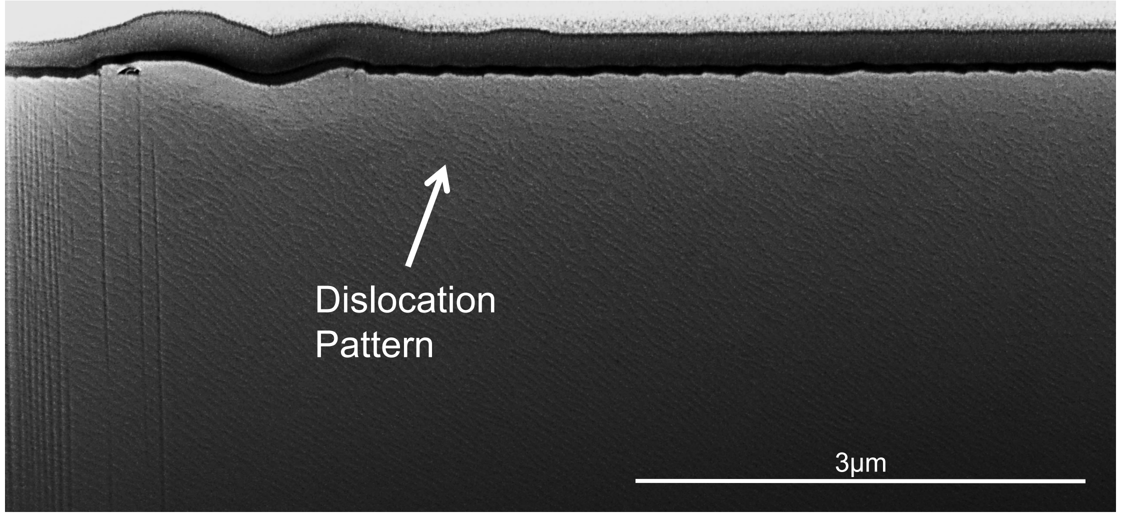
On a SA system sample





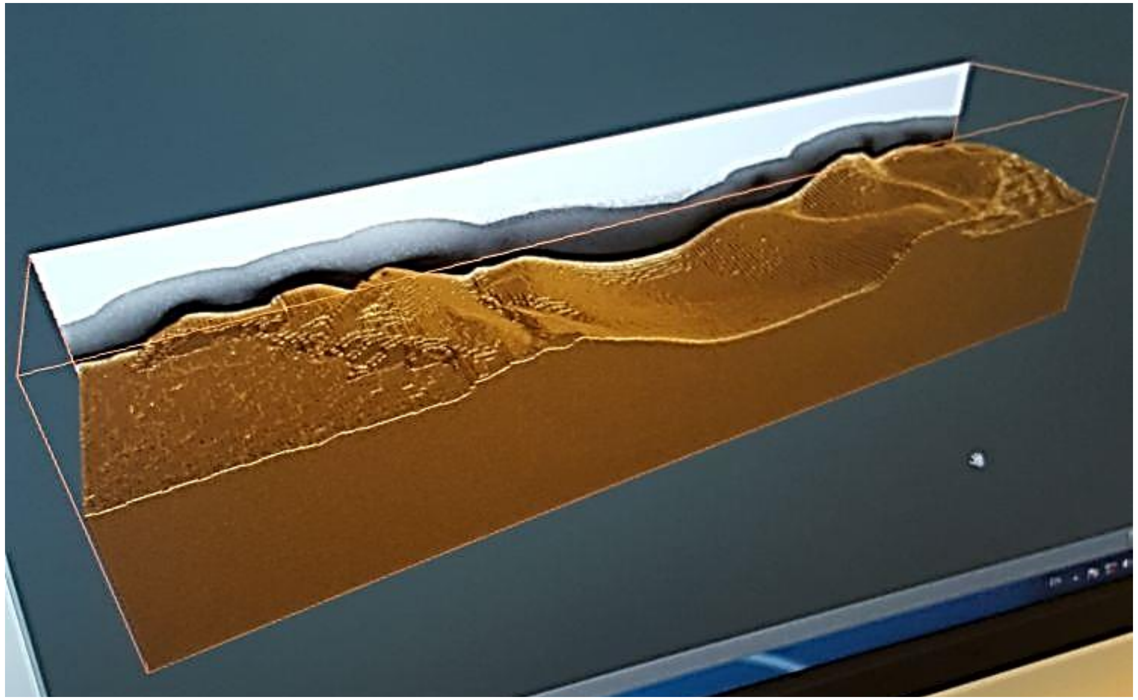






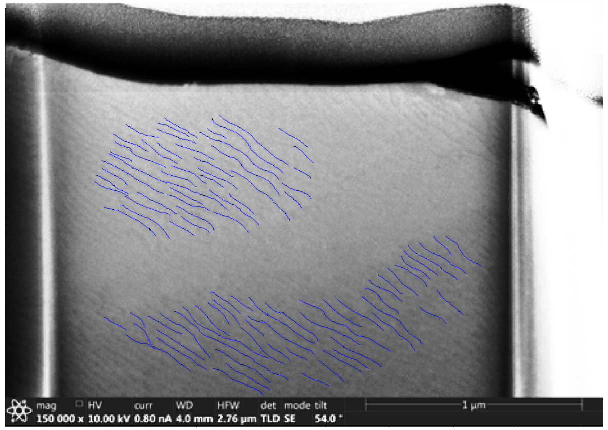
The dislocation pattern is visible when FIB milling is perpendicular to terraces observed on the surface

Tomography at a Break-Down Site

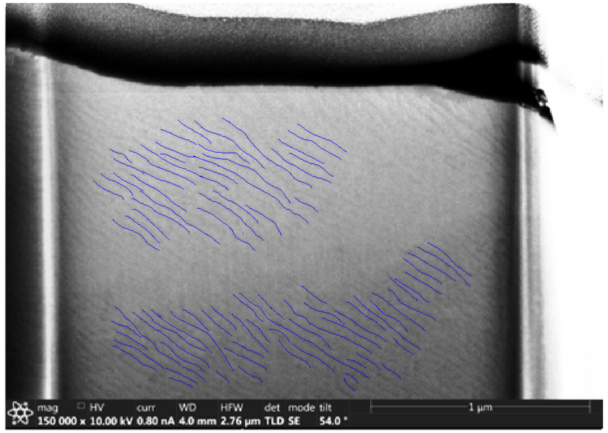


FIB cross sections that were taken with steps of 5nm from each other

Subsequent images:



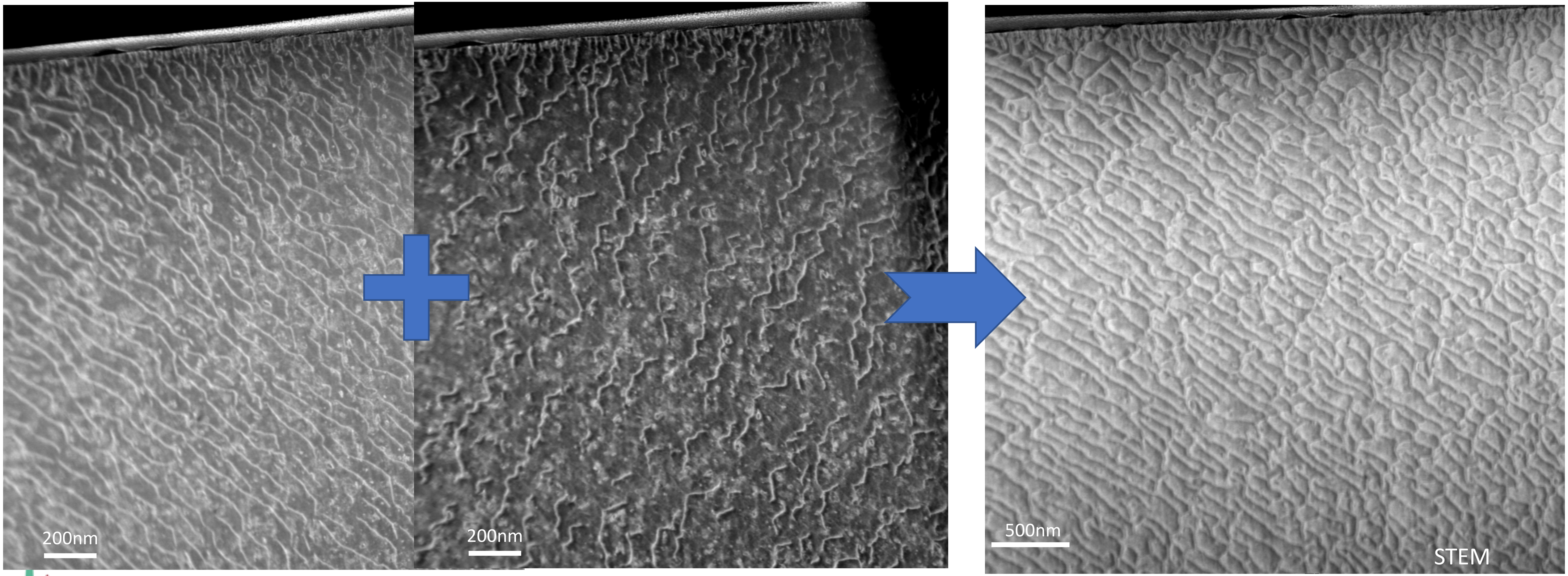
→



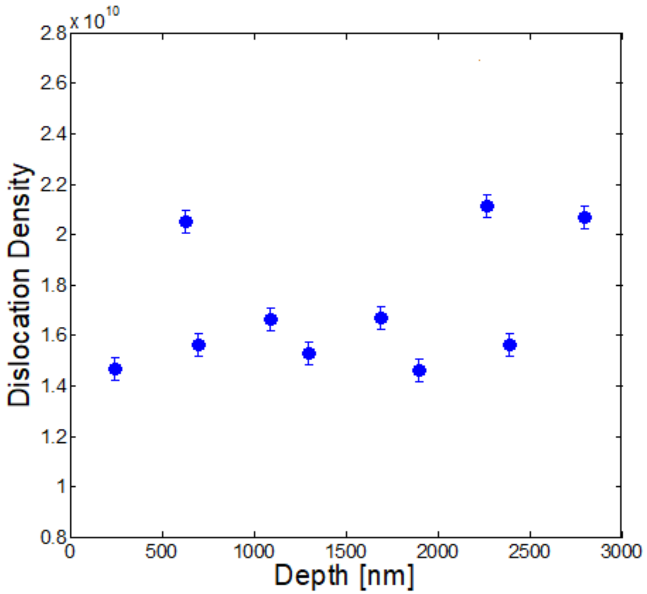
The dislocation pattern changed in each step

Observed dislocation structure – super position of various systems

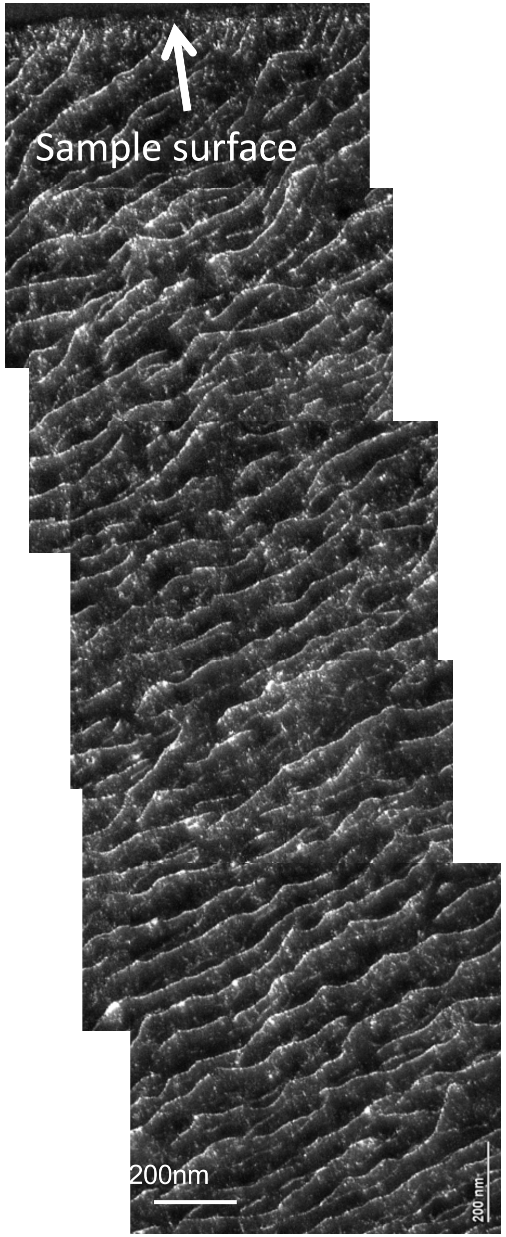
TB 200 TB 111



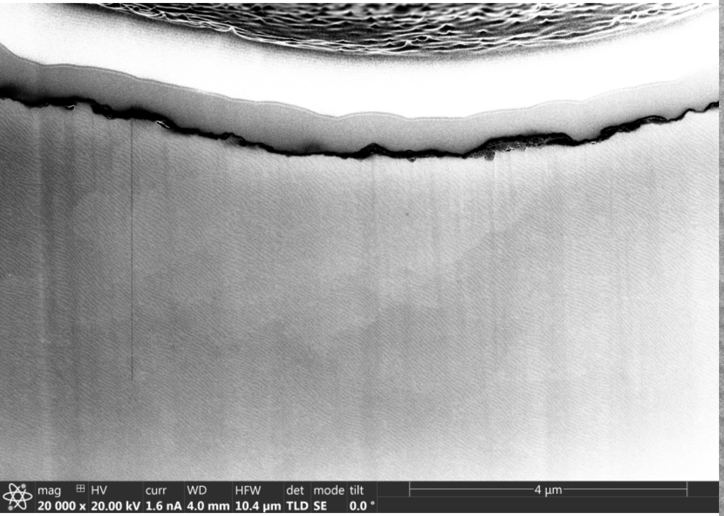
- We do not observe significant variation of dislocation density with depth.

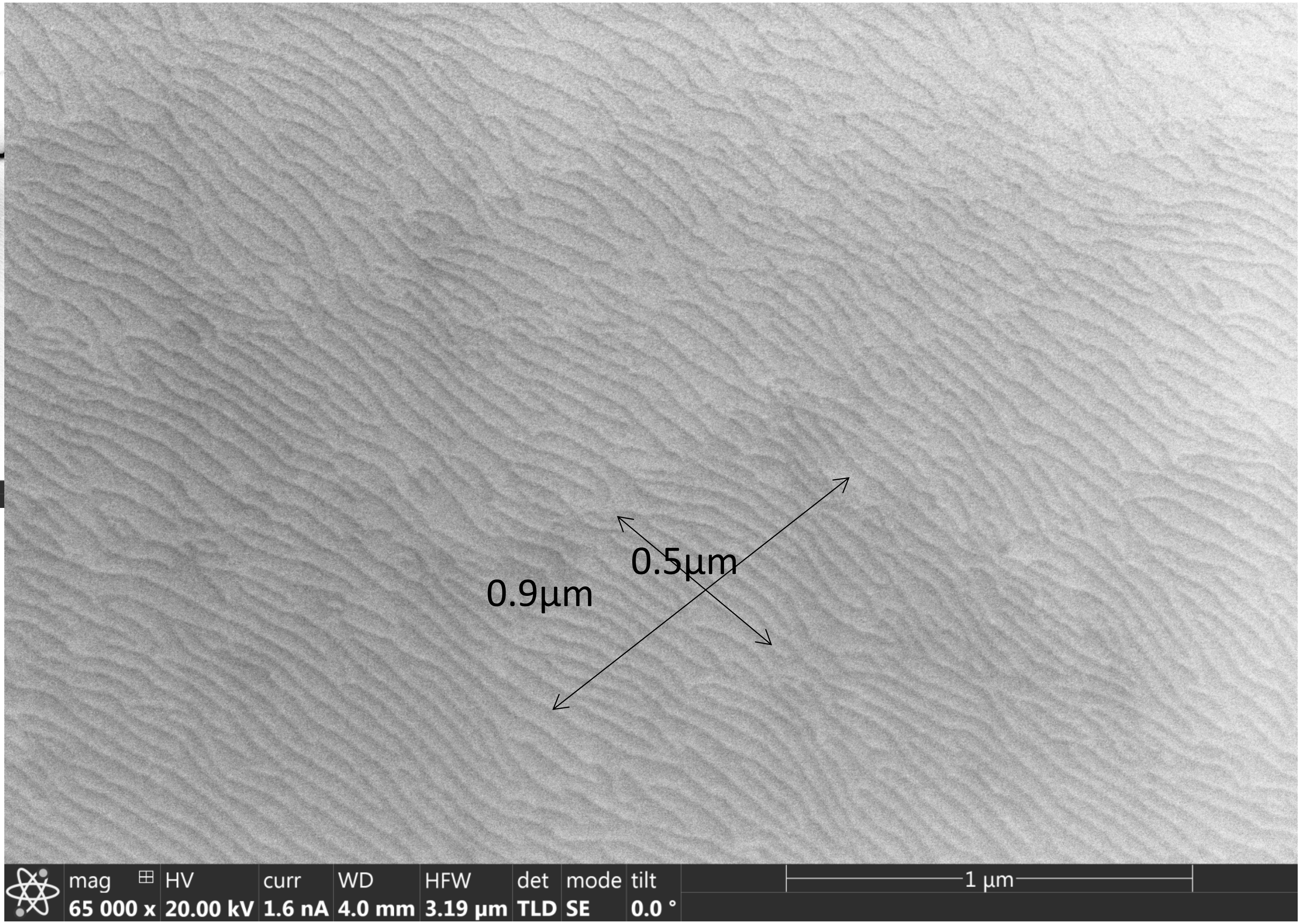


RF 23 CS near the iris



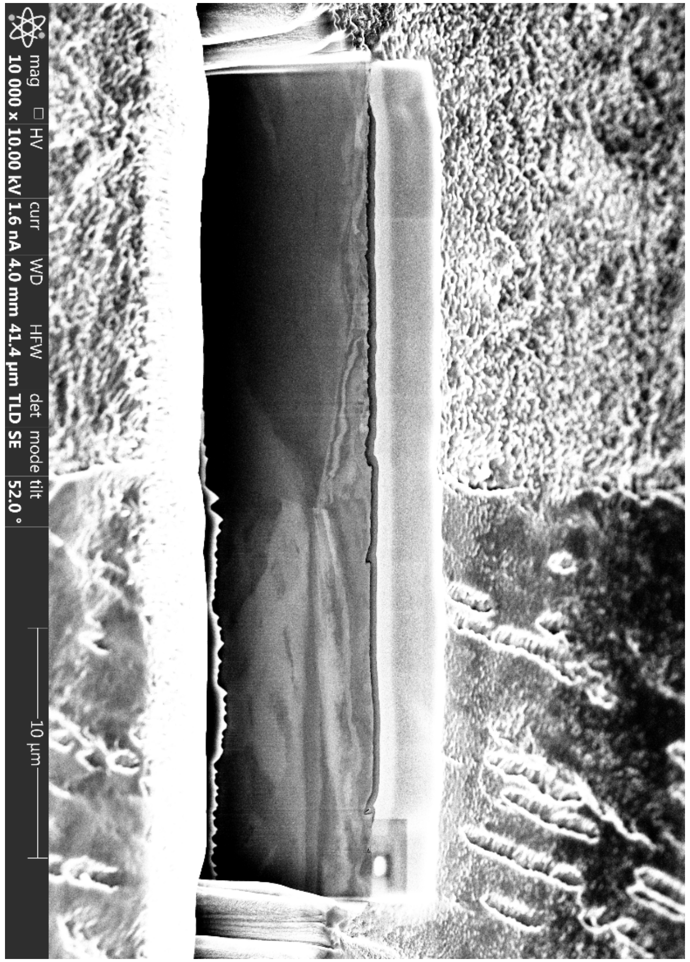
Estimating correlation length

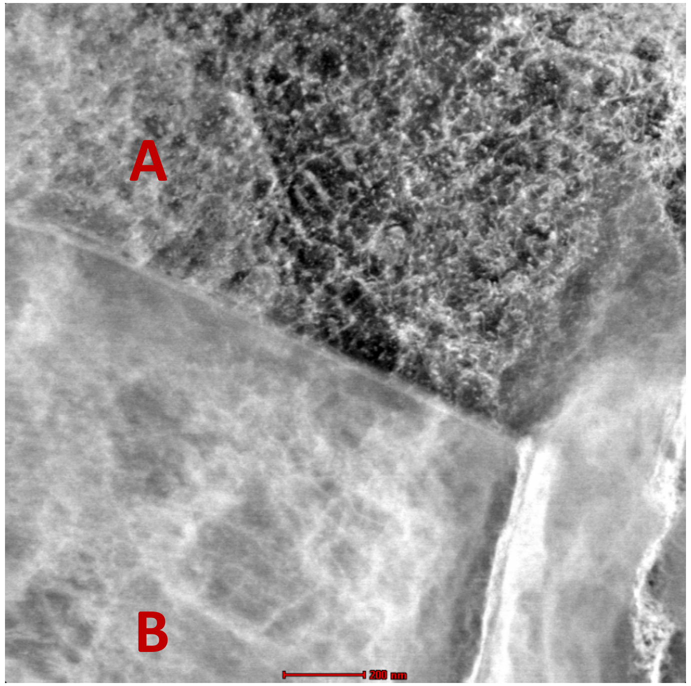


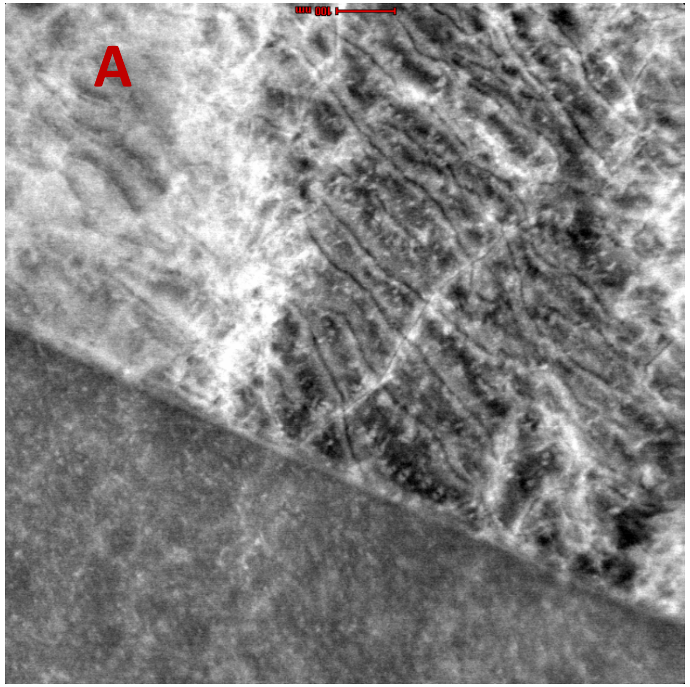


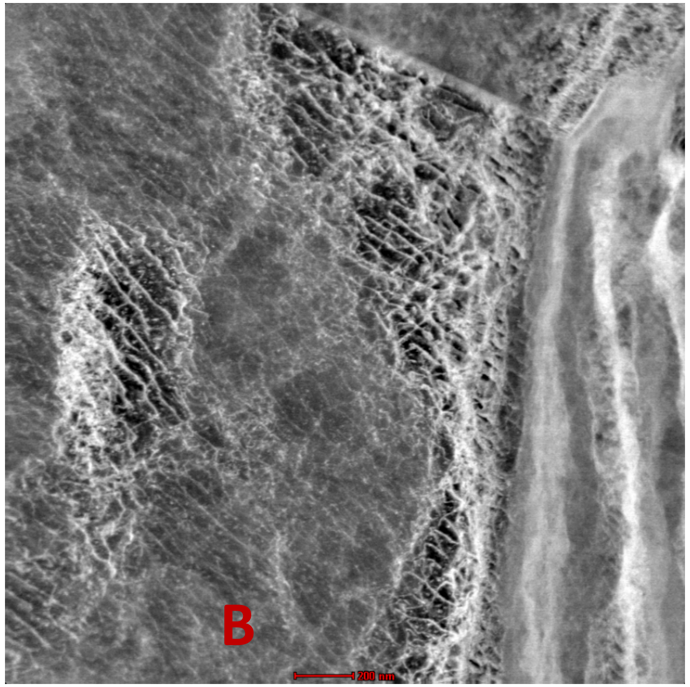
- Minimal ~1micron; up to 10 microns (ebsd)

Hard Cu-Ag electrode… similar underlying structure



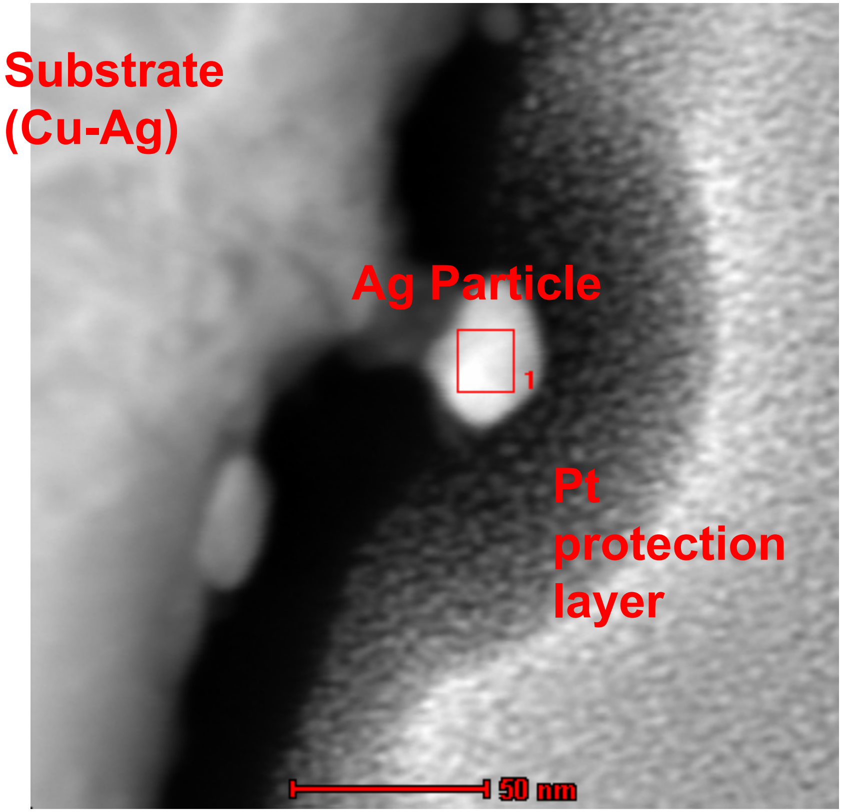




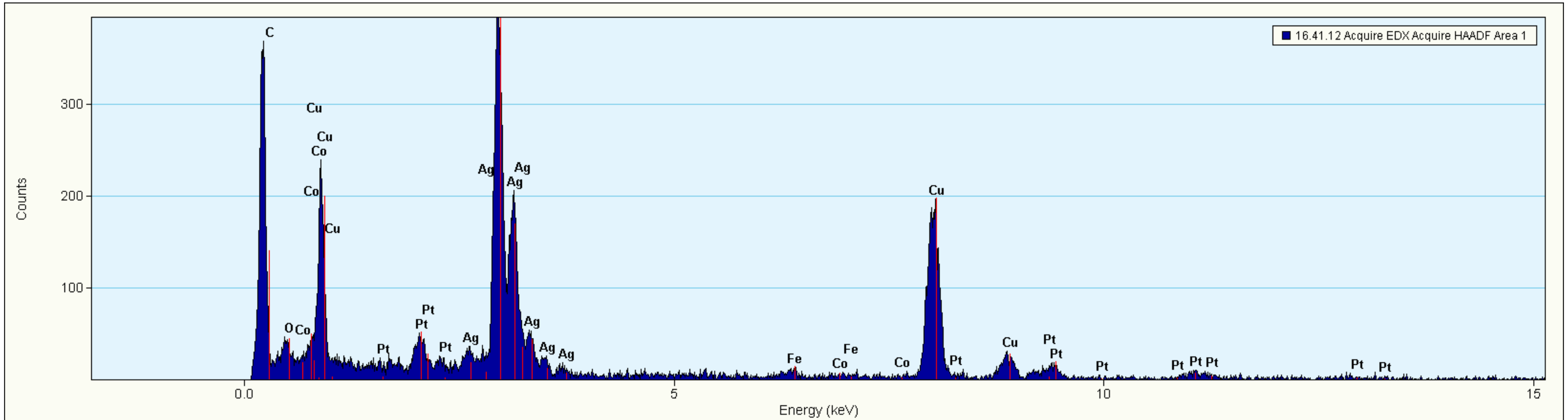


Observable dislocations on both sides.
Decreased upper limit on correlation length

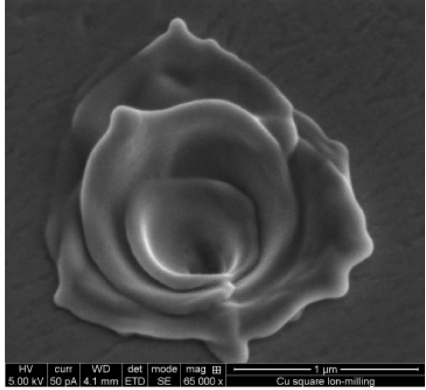
Hard Cu-Ag – TEM



CuAg (0.08%) -
Ag precipitate to the surface



Constraining models and possible predictions



- Microscopy set limits on model - but not exact.
  Picture is consistent – but not not conclusive.
- Current model – conclusive microscopic test – impossible!

Crytical Experimental scenarios

Microscopy – verify and consistency (conditioning , performance correlation)

Direct probing of dynamics:

- Fluctuations in dark currents
- Acoustic signals from mobile dislocations

Testing predictions:

- Temperature dependence.
- Structural effects

Temperature dependence – the need for cryogenic system

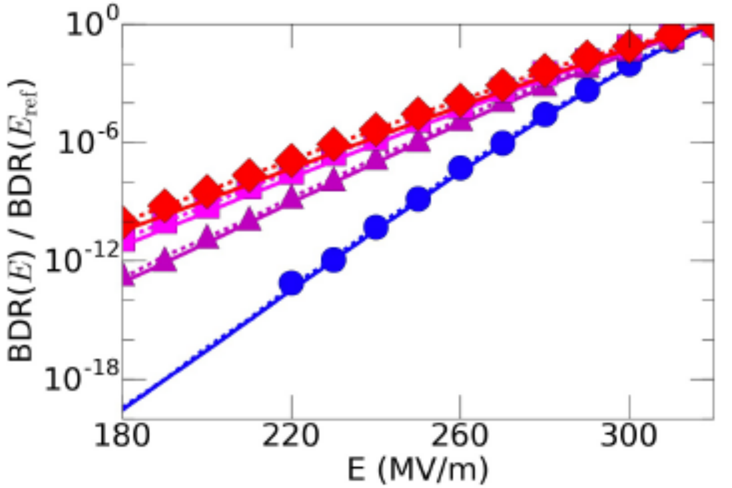
Model specific predictions:

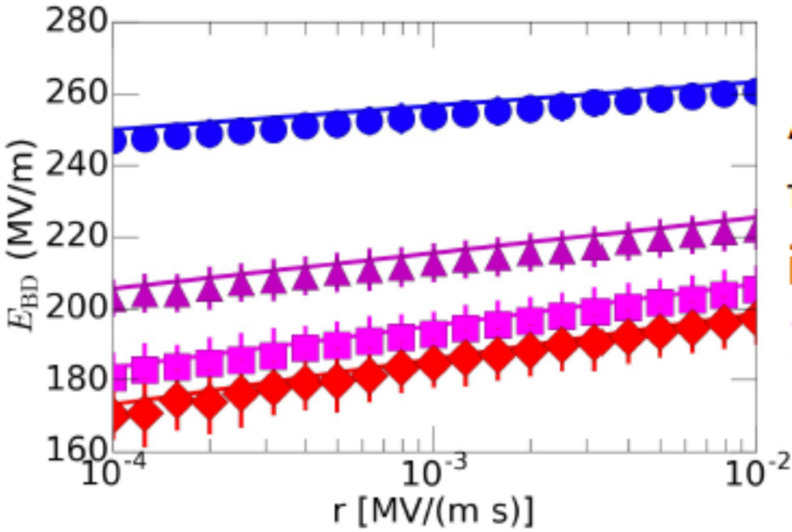
- ”Classical” scenario Temperature effect on BDR versus field slopes
- Dynamic specific - ramping up field at various rates. Average “field for BD”

Hopefully all to be tested soon

*we will certainly re-calibrate the model….
(but hopefully not abandon it)

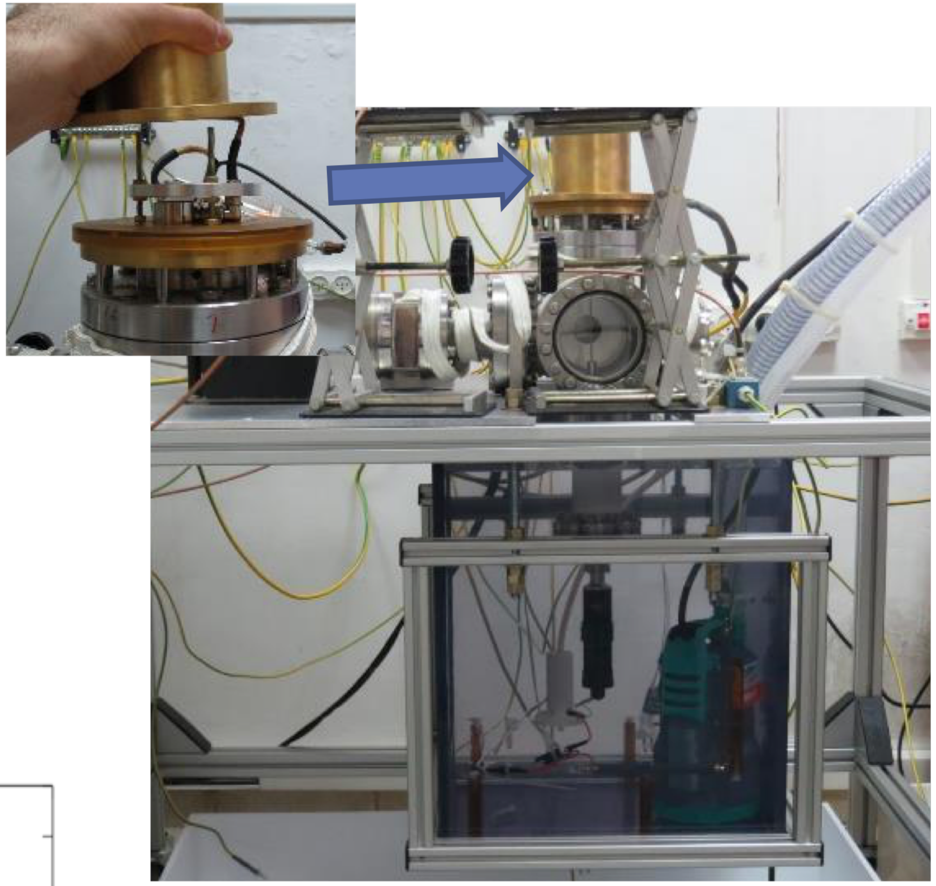
BDR as a function of the field at 100 K, 200 K, 300 K, and 400 K.

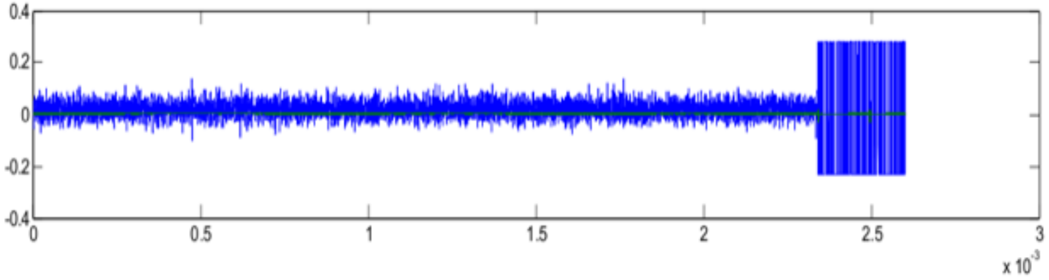




Even more critical…
Acoustic emission measurements (under development)

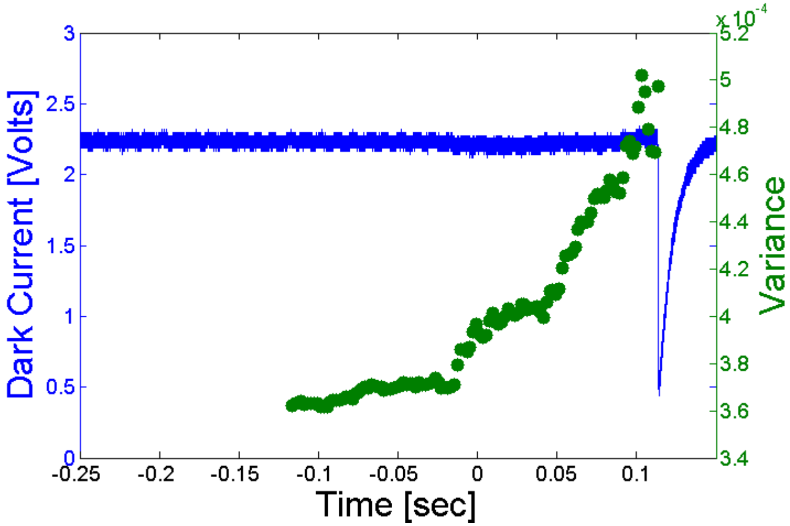
- Acoustic emission – distinctive signal from moving dislocations.
- Hope to correlate dark currents and acoustic signals
- Identifiable pre-BD fluctuations?
- WE claim that conditioning is due to plastic modifications of the surface- can we form ”well informed” conditioning?



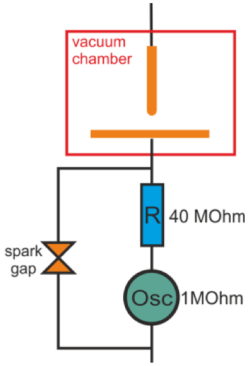


Pre BD fluctuations…

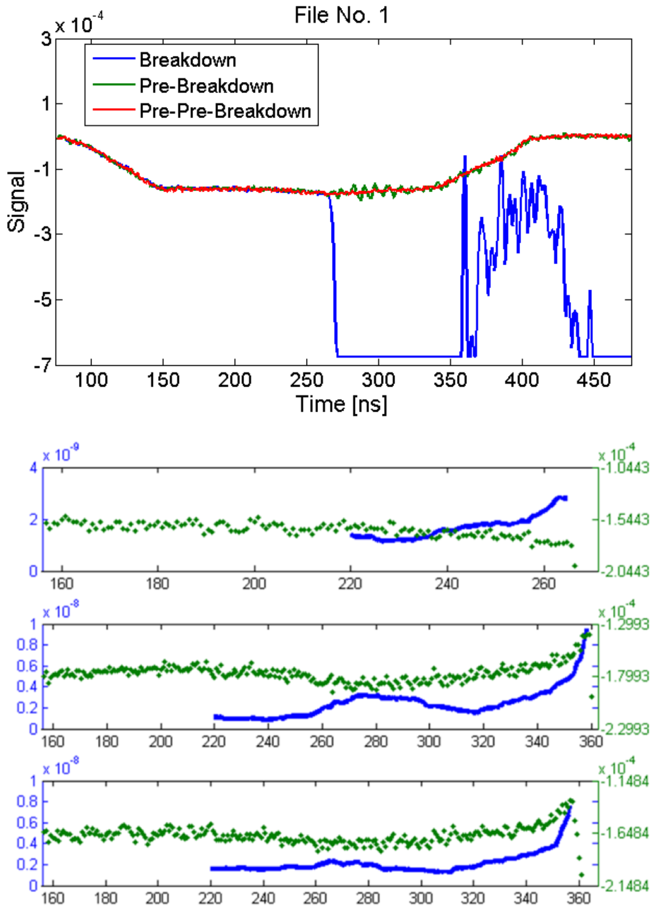
- Prior to critical transition, we expect increase in fluctuations.
  DC and RF indications of pre-breakdown increase in dark current variance



DC data –
Iaroslava Profatilova
Tomoko Muranaka



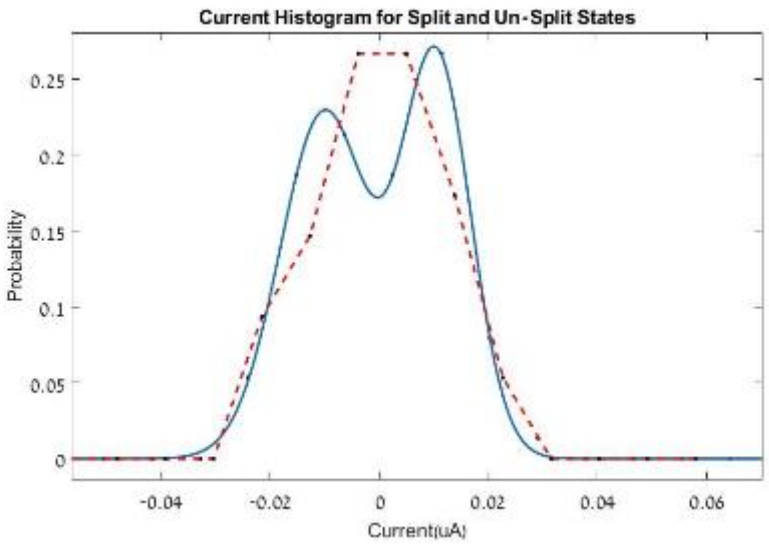
Need for :
High resolution at peak
+ dt < 1 nsec (f>2Ghz)

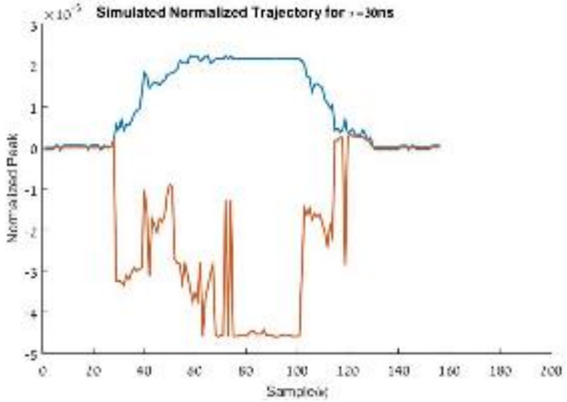


RF data - Alberto Degiovanni

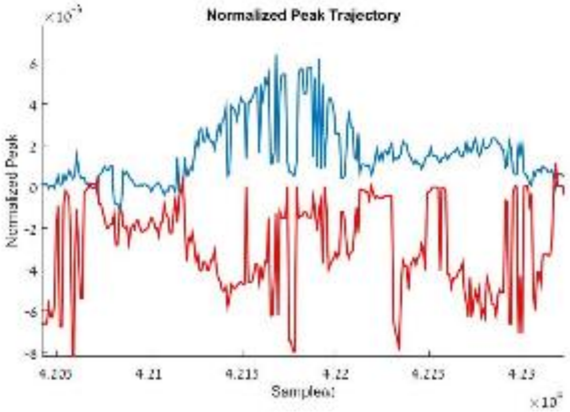
Dark current distributions

- Dark currents are expected to have a Gaussian distribution.
- High frequency (GHz) data sets(from DC setup) demonstrate “splitting” to two Gaussians.
- “life time” of ~10-50 nsec





Simulated signal



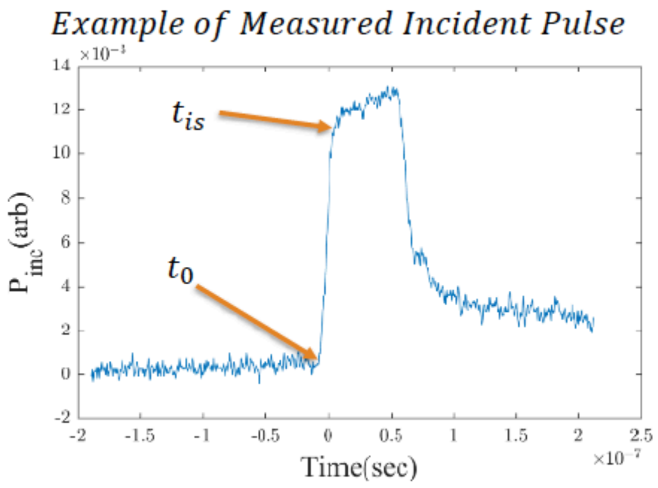
Measured signal

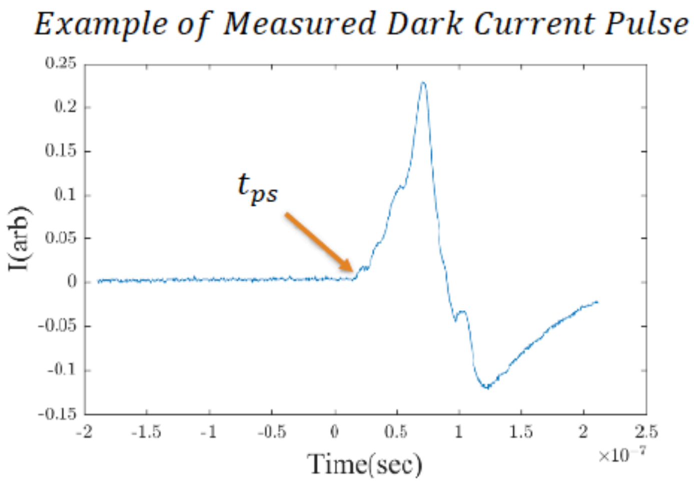
Need MUCH more GHz data!

Sagi Lachman
data by Tomoko Muranaka

Possible increased fluctuations in dark currents

(Sagy Lachmann and Jan Paszkiewicz)



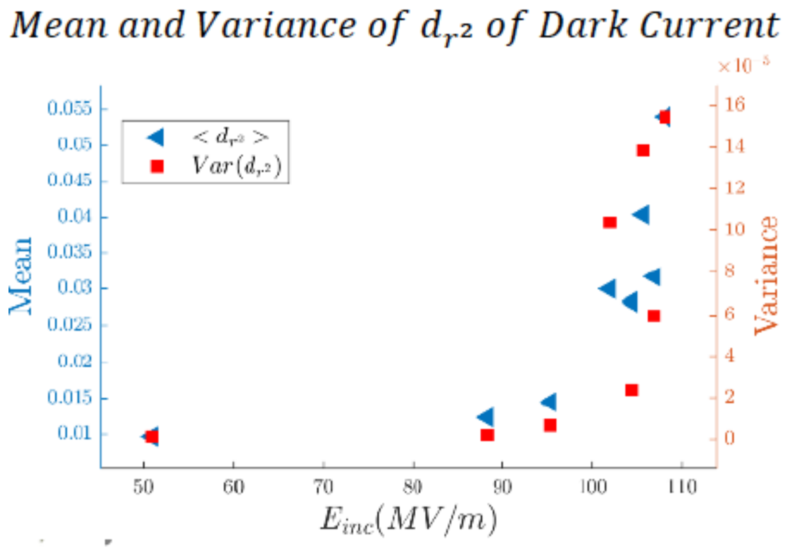


- For a set of N measurements vectors of the same incident power $a_i^j, j = 1 \ldots N$ the mean pulse is defined as:

$$e_i = \frac{1}{N} \sum_{j=1 \ldots N} a_i^j$$

- The variation of each vector is defined as:

$$d_{r^2}^j = \sum \left(a_i^j - e_i\right)^2$$



Looks promising,
But still trying to figure out the role of amplified noise

How can we do better?

Ultrahigh Strength and High Electrical Conductivity in Copper

Lei Lu, Yongfeng Shen, Xianhua Chen, Lihua Qian, K. Lu*

Science, 304 (2004) page 422

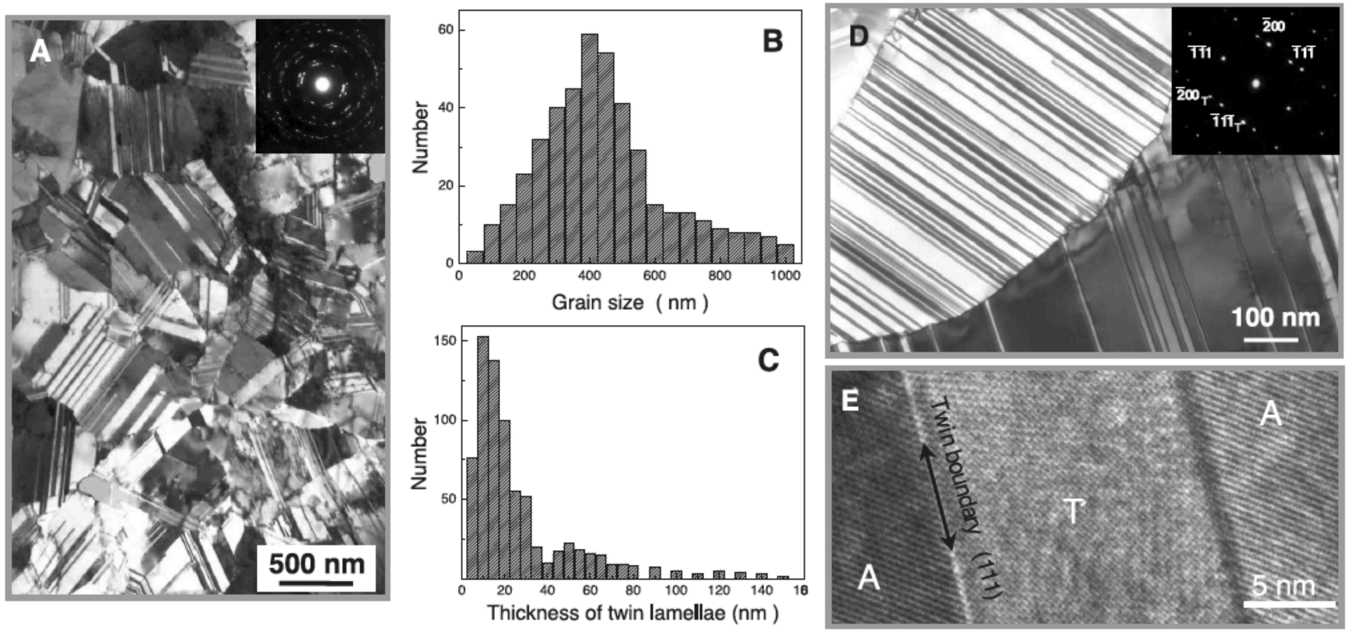


Fig. 1. TEM observations of the typical microstructure in an as-deposited Cu sample. A bright-field TEM image (**A**) and the electron diffraction pattern (inset) show roughly equiaxed submicrometer-sized grains with random orientations separated by high-angle GBs. The statistical distributions for grain size (**B**) and for thickness of the twin/matrix lamellae (**C**) were obtained from the many TEM images of the same sample. Electron diffraction patterns [inset in (D)] indicate that the twins in each grain are parallel to each other in {111} planes (**D**), and high-resolution TEM images (**E**) show that the twins follow a sequence of ATATA. . ., with twinning elements, for example, A: $(\bar{1}1\bar{1})/[\bar{1}12]$ and T: $(\bar{1}11)/[1\bar{1}2]$.

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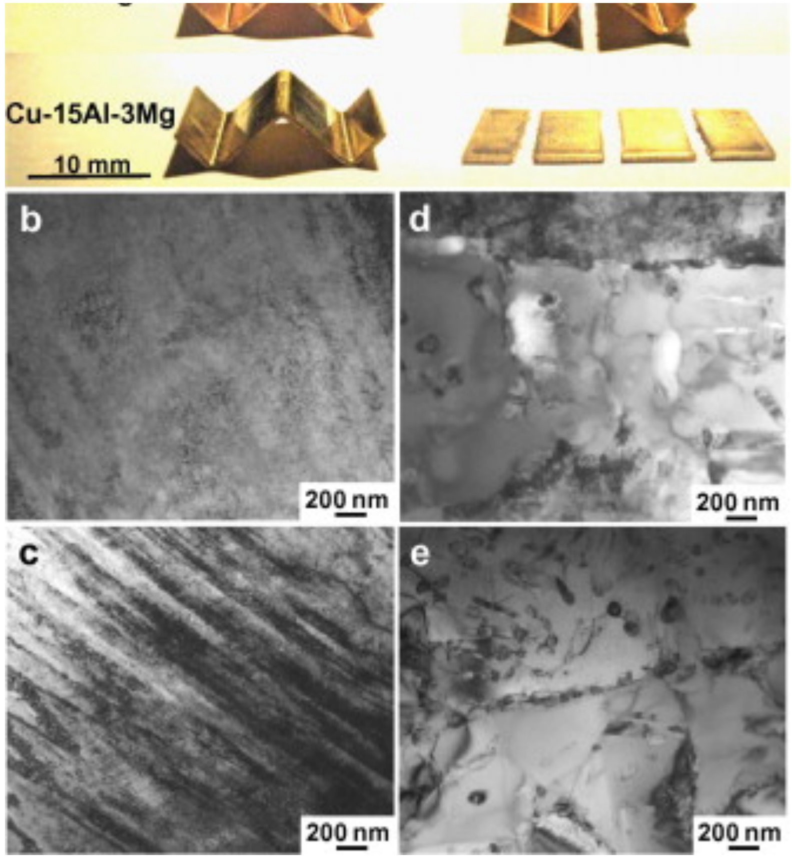
Scripta Materialia 68 (2013) 777–780
www.elsevier.com/locate/scriptamat

Solid-solution copper alloys with high strength and high electrical conductivity

Kazunari Maki,* Yuki Ito, Hirotaka Matsunaga and Hiroyuki Mori
Mitsubishi Materials Corporation, Central Research Institute, 1975-2, Shimoishitokami, Kitamoto, Saitama 364-0022, Japan



Summary and Outlook

- Proposed model – stochastic in time and position.
  consistent with microscopy
- Identified a possible controlling microscopic dislocation density
- Possible intrinsic conditioning mechanism – surface hardening
- A set of upcoming critical experimental scenarios –
  cryogenic temperature (Upsala?), fluctuations(cern+huji), AE (huji).
- Theoretical challenges – post transition ignition (Djurabekova group), improved critical transition model (huji)
- Where can this take us:
  - improved control over material effects (design?!)
  - Optimized conditioning
  - Warning signals?