The Harvey M. Krueger Family Center
for Nanoscience and Nanotechnology

האוניברסיטה העברית בירושלים
THE HEBREW UNIVERSITY OF JERUSALEM

Observations on the link between cathode plastic activity and arc nucleation

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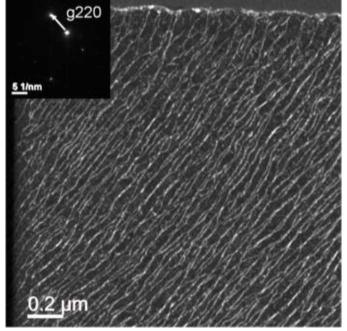
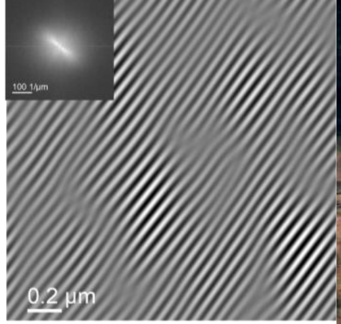
PAZY
EXCELLING IN SCIENCE

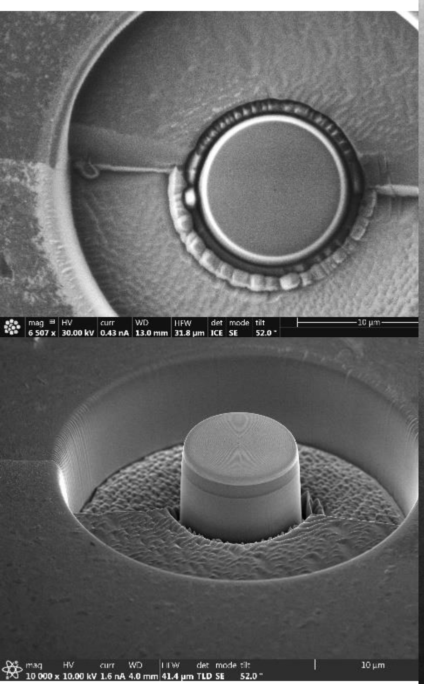


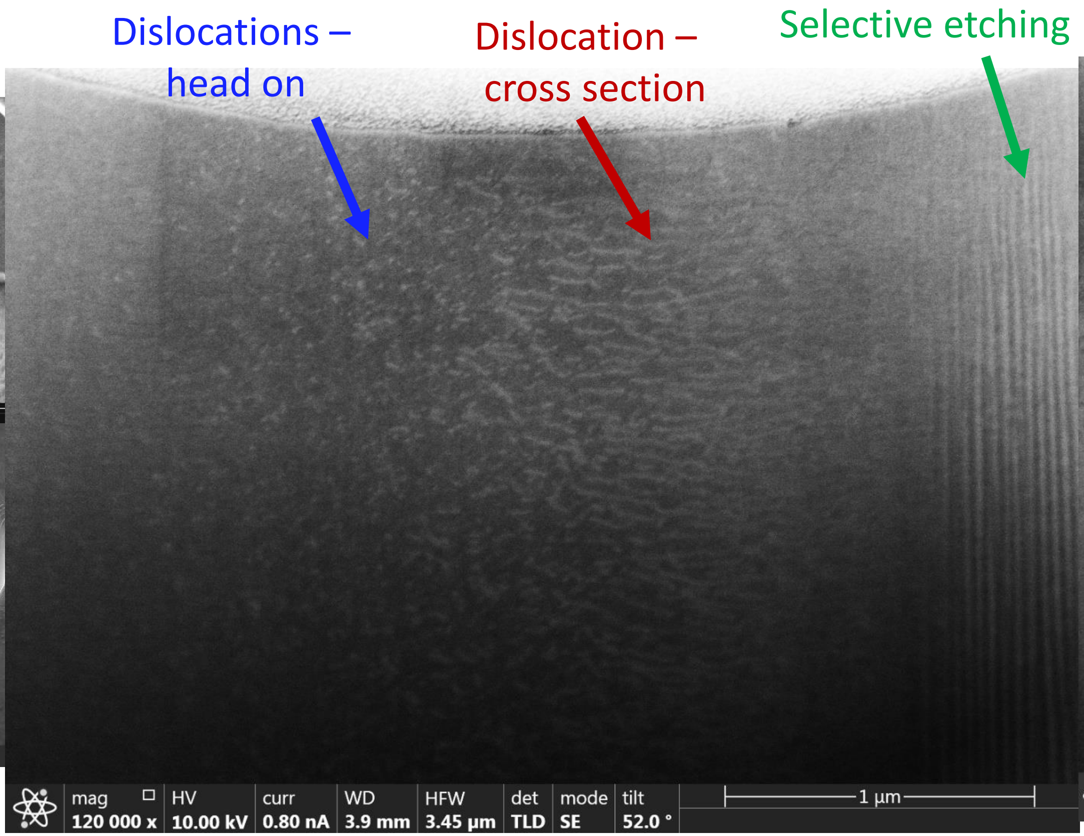


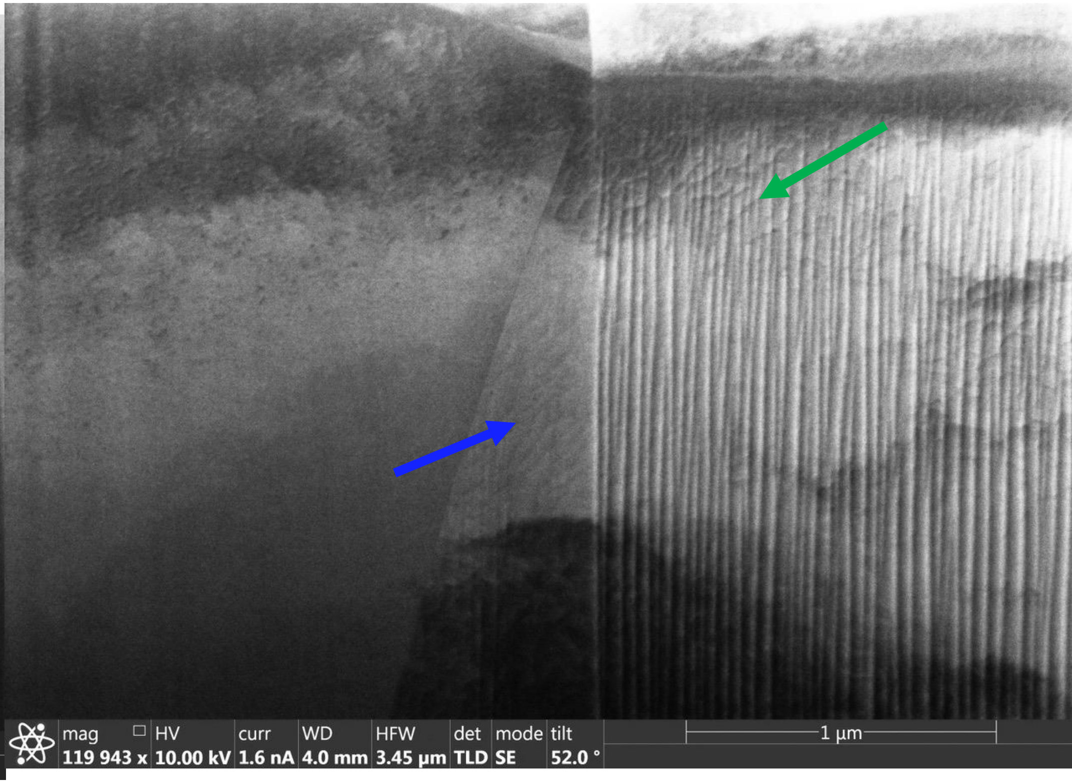
outline

- Breakdowns due to dislocations "stampedes"
- Microscopy of electrodes – identifying the zebras
  - Demonstrating universal sessile dislocation array in Cu electrodes
  - Can we identify distinct conditioning effect?
- Modelling the zebras response
  - Mean field model for **f**luctuations in **m**obile **d**islocations response (FMD)
- From stampedes to BD
  - Stampedes – so what?
  - Identifying pre stampede – even without explicit link to BD

Observing dislocations





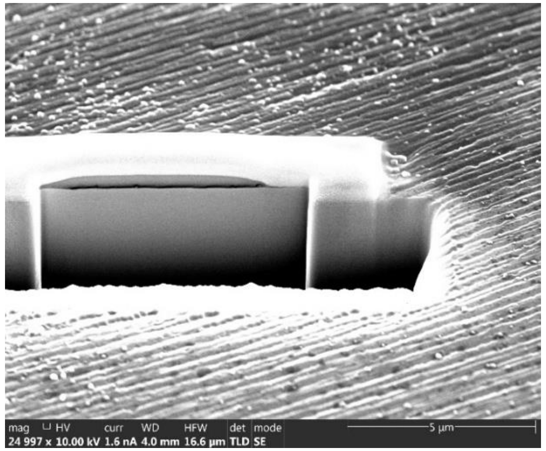


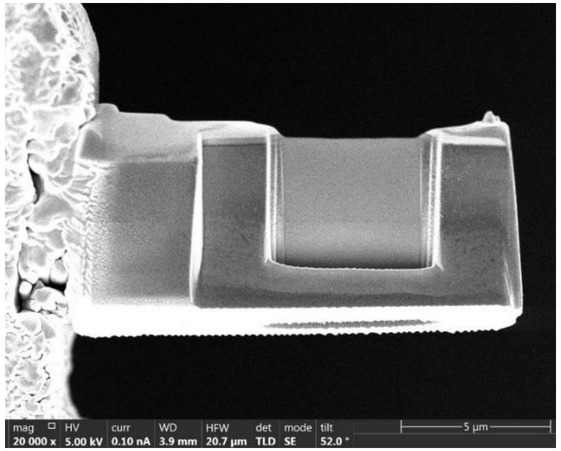
- Visibility conditions – cross-section orientation
- Curtaining under FIB – surface dependent

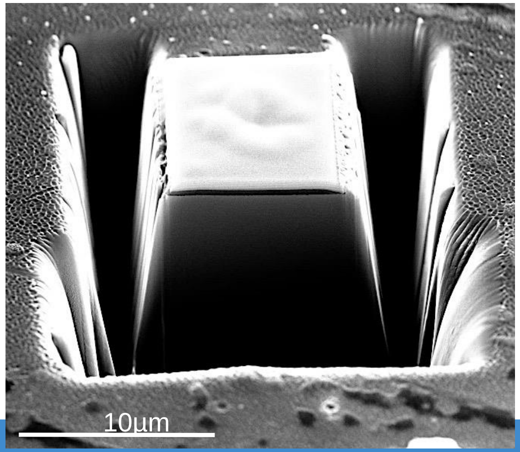
Organized array of dislocations

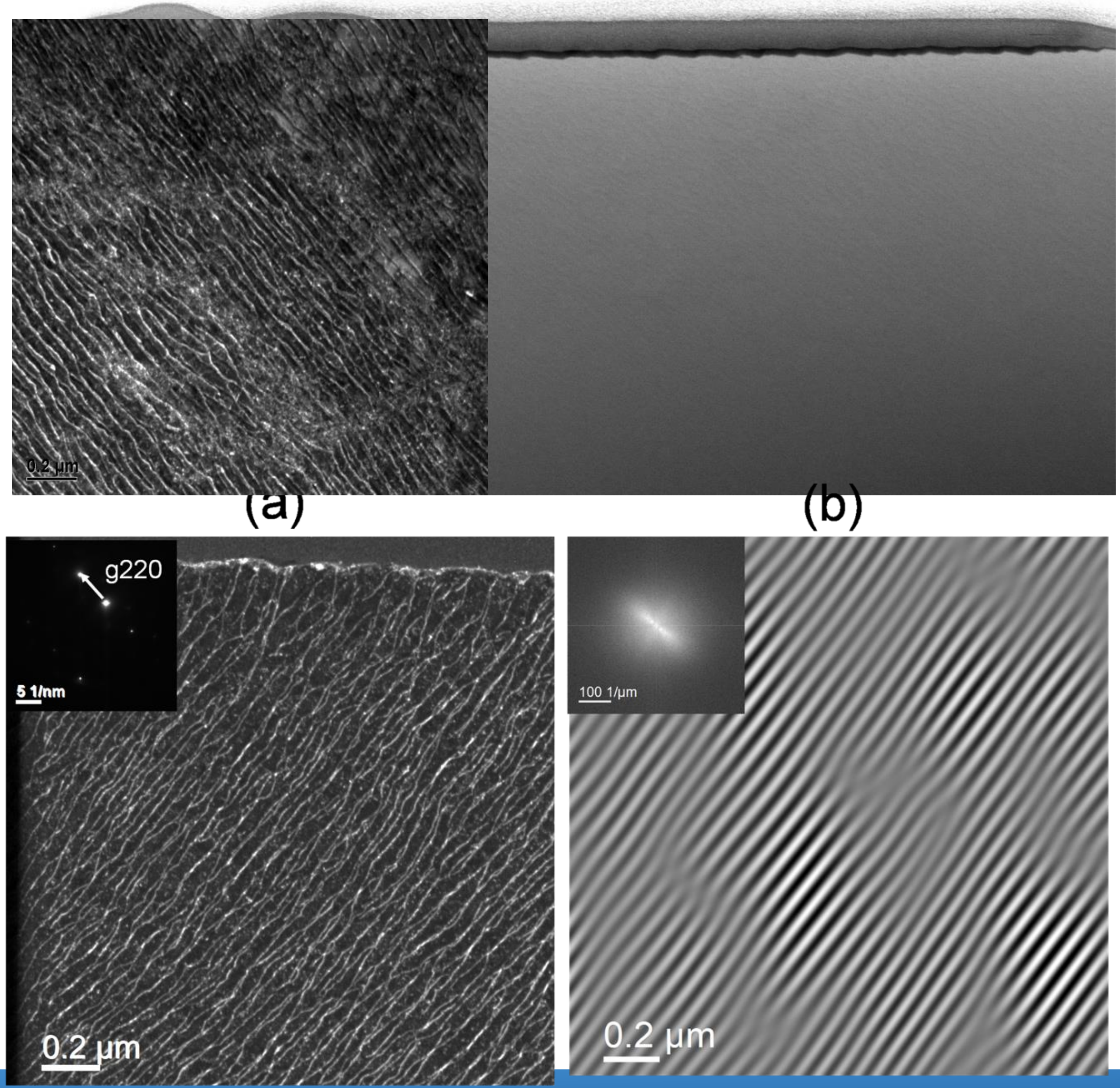
Cut below surface to estimate dislocations structure using SEM

Using Fib –create top or side view lamellas for TEM and STEM



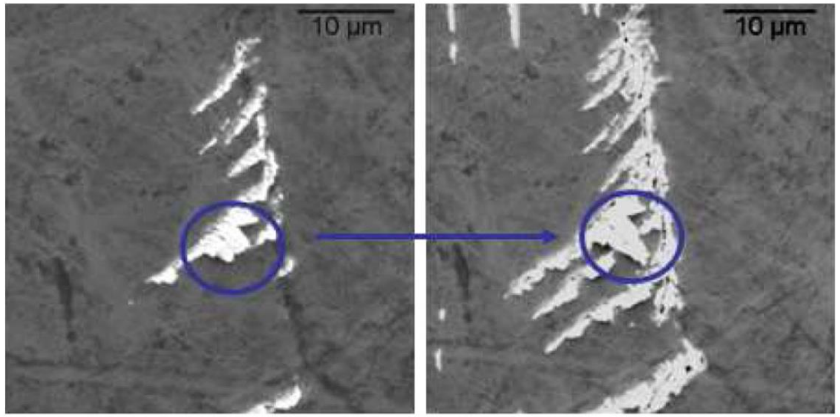




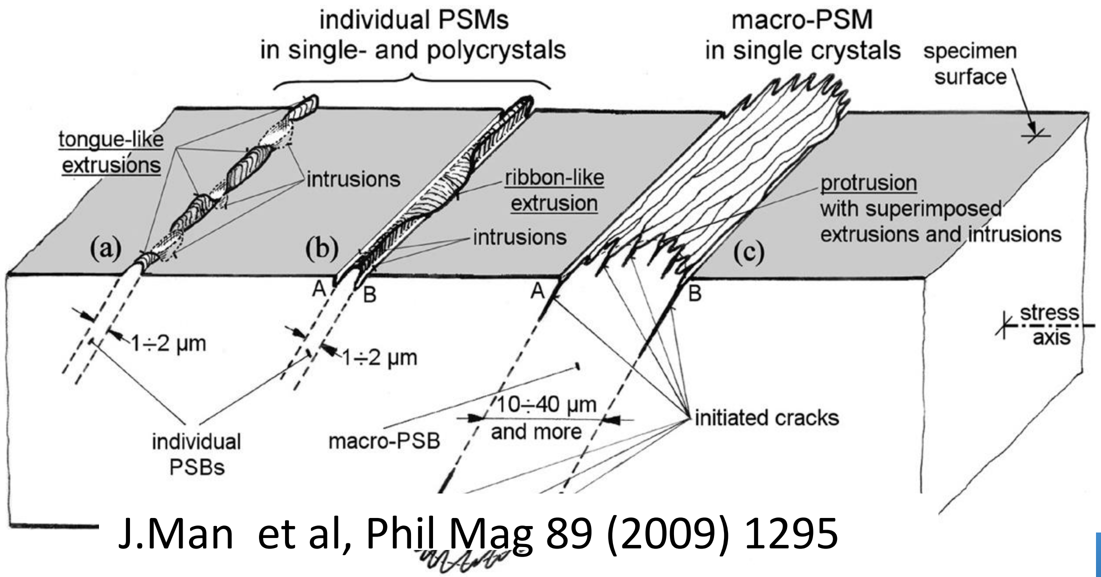


Dislocations are known to create persistent slip bands and protrusions

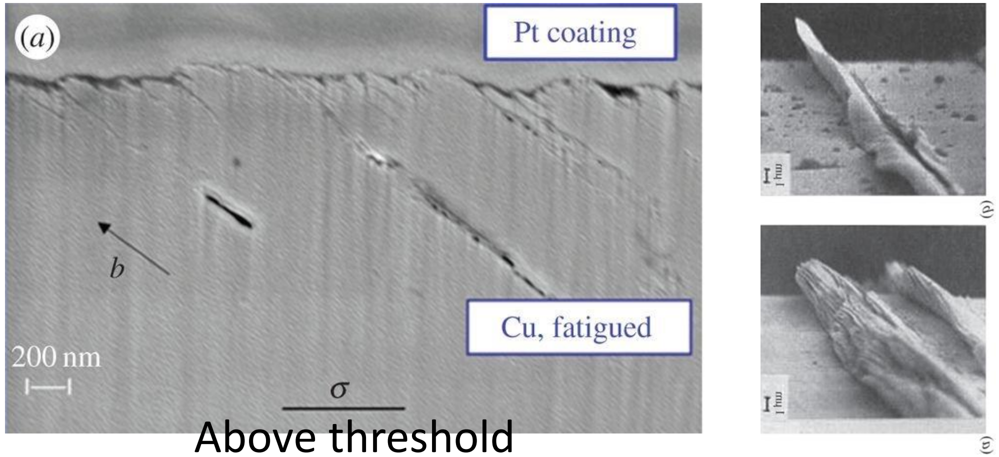
- Previously observed on fatigued surfaces.
- Significant sub-surface PSB leading to surface features.
- Stochastic response at sub-yield stresses.
- PSB exist in various scales – down to 10 nm. These can lead to sudden increase in current



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



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



Above threshold

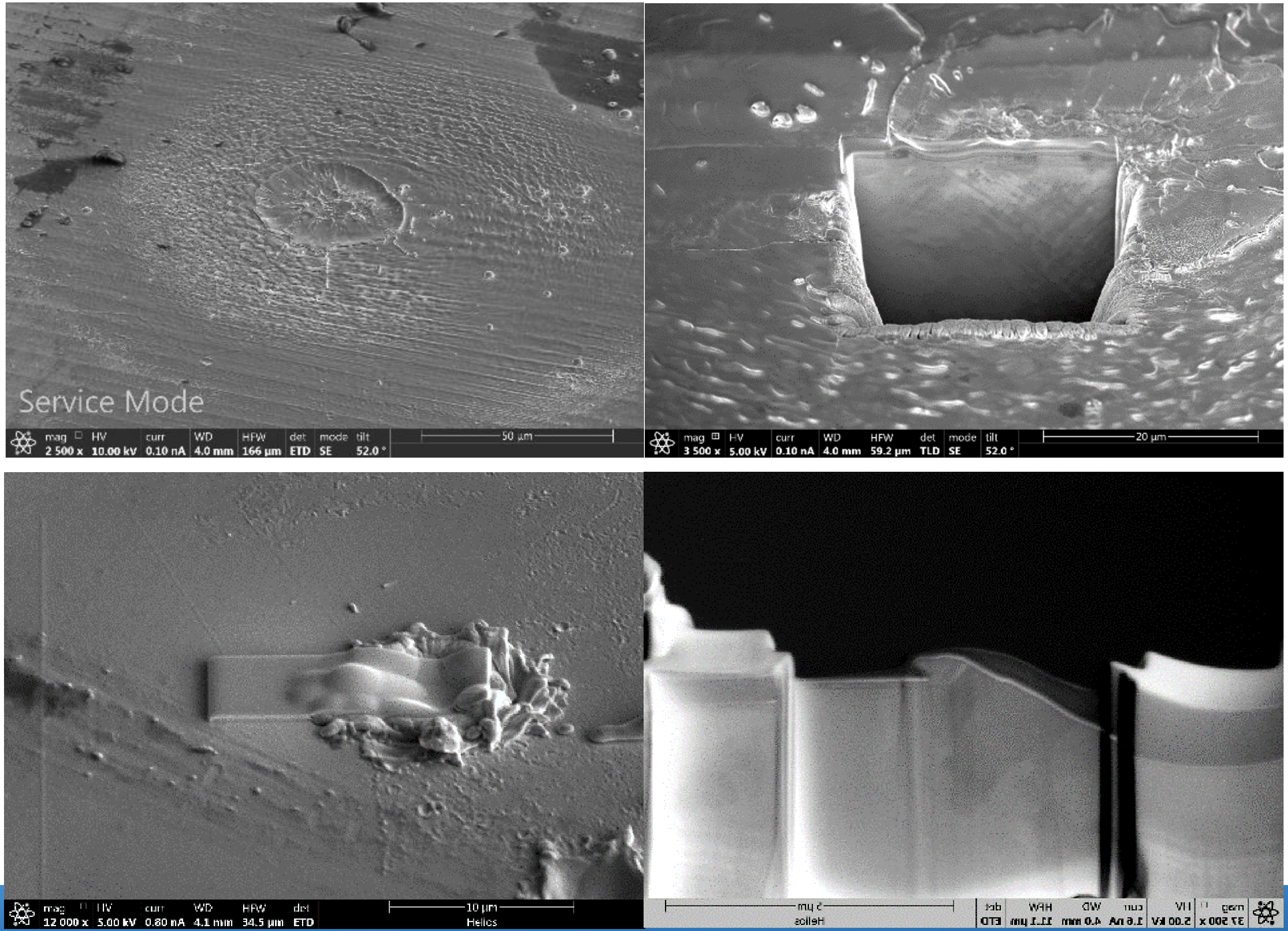
Polycrystal Cu - fatigued ~10^{10} times sub PSB threshold
Haël Mughrabi Phil. Trans. R. Soc. A 2015

Microscopy of BD events (RF and DC samples)

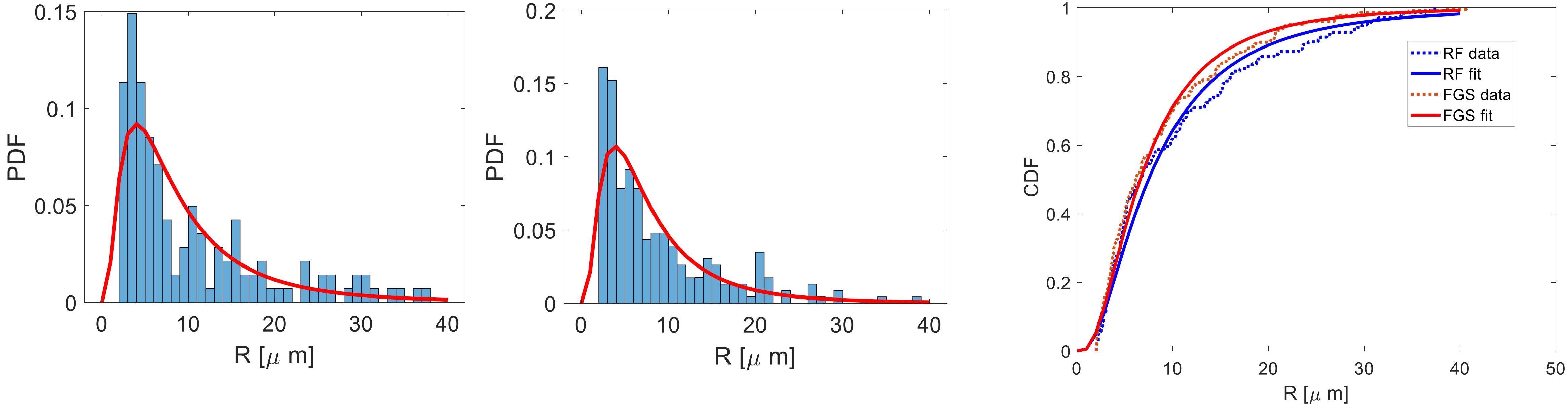
BD are a result of copper plasma formation which creates significant plastic activity at BD site

BD craters can be small or engulfed in large pools of melted copper

But in general the remnants of this violent events do not hold info on what preceded them…



Note: DC(FGS) – RF correspondence

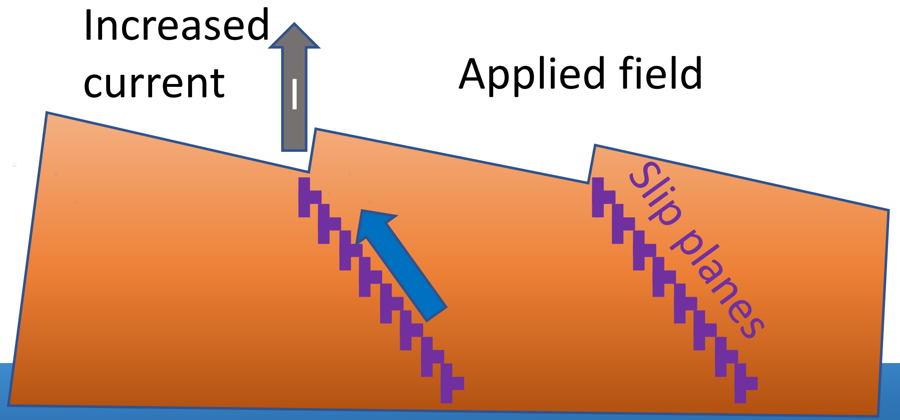


- Similar BD craters.
- Main difference –
  "Liquid pools" attributed to post BD evolution

Modelling Breakdowns as rare critical events

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

- Underlying assumptions ("knowns"):
  - BD are formed due to rare localized amplification of thermionic emission which leads to emission of neutrals and seeds plasma formation
  - BD involves plasma evolution and surface sputtering.
- Our main hypothesis:
  - Intrinsic breakdowns are initiated due to a critical plastic process.
  - These are driven by collective dislocation motion below the surface which leads to subsequent surface modifications.

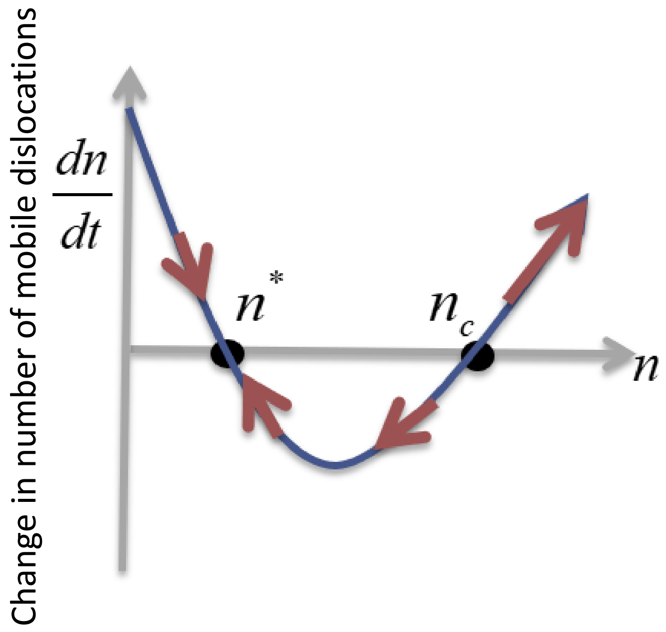


Stochastic model

- Observations: dense ordered sessile array of dissociated dislocations (stabilized by elastic interactions) .
- Under appropriate drive - Such arrangement can become mobile…
  We created a mean field model describing evolution of the mobile dislocation population:

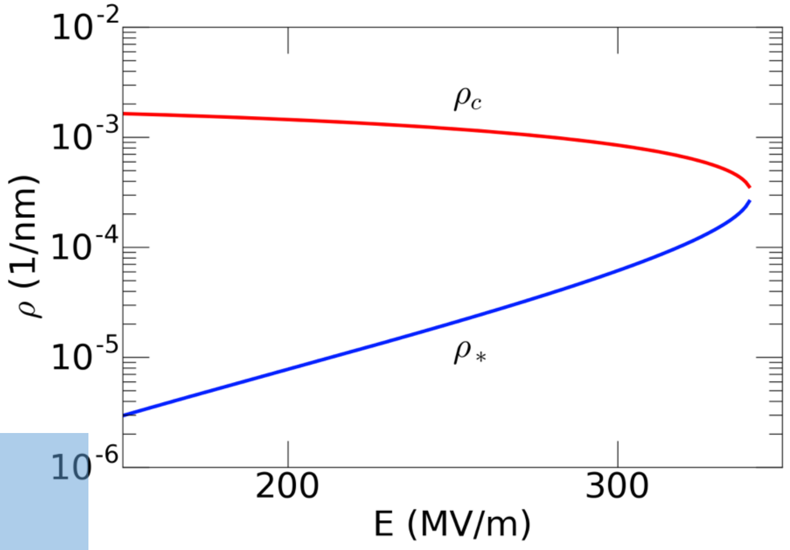
PHYSICAL REVIEW LETTERS **120,** 124801 (2018)

Stochastic Model of Breakdown Nucleation under Intense Electric Fields



Governing equations:

- Increase in mobile population – interactions with field and moving dislocations.
- Arrest due to collisions
- Cooperative critical transition in mobile dislocation population generates nucleation event



We propose that this transition
 - start of a run away in mobile population -
can lead to a nucleation event through its effects on the surface

Proposed observations

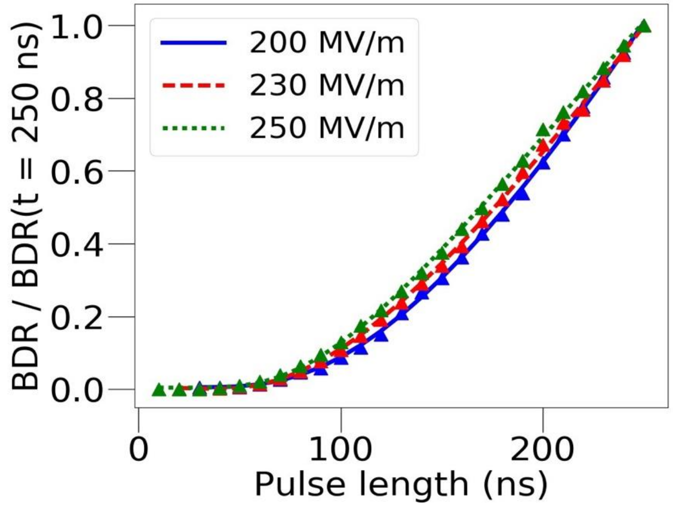
Temperature dependence :

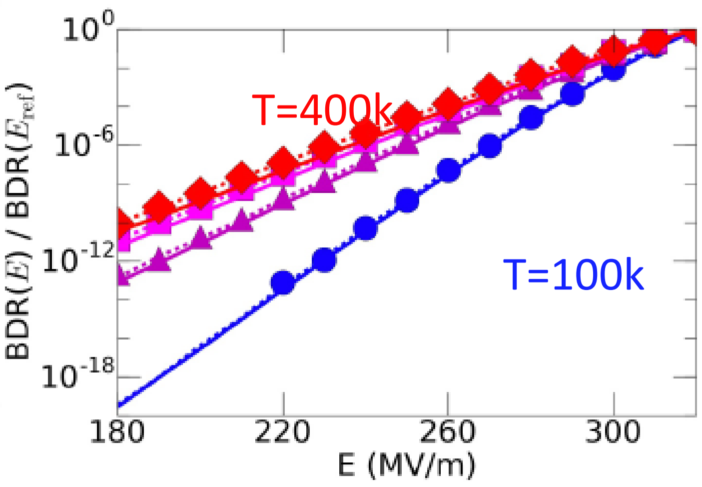
- Need for Low T
- ”Classical” scenario: Temperature effect on BDR versus field curves.
  Dynamic - Ramping up field at various rates. Average “field for BD”
- T dependence – would lead to verification of activation temperature and kinetics.

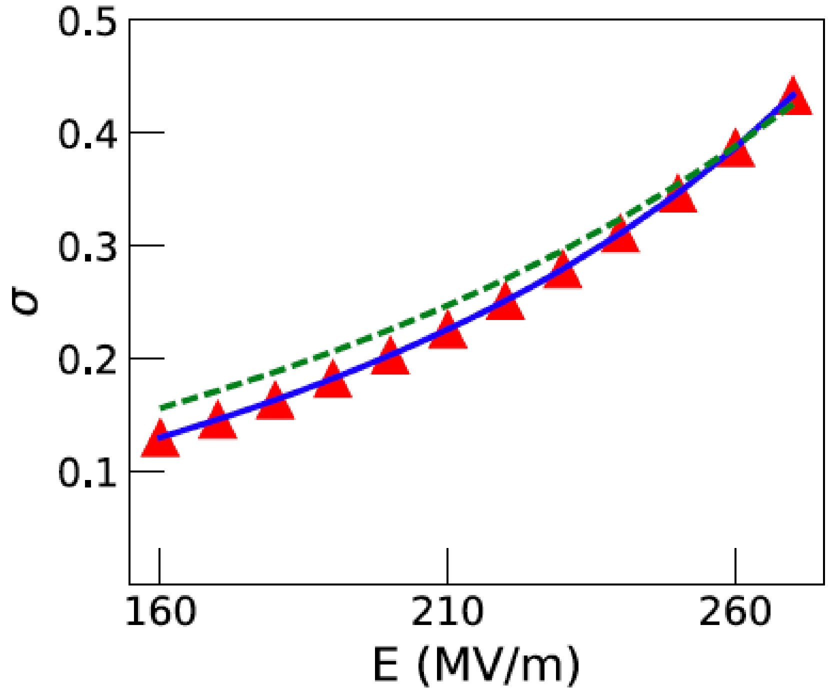
Time dependency - Non Linear regime.

But most important – prior to BD as field is increased fluctuations in the population - and the dark current should be observed!

See Eli’s talk on Wednesday!







PHYSICAL REVIEW ACCELERATORS AND BEAMS **22**, 083501 (2019)

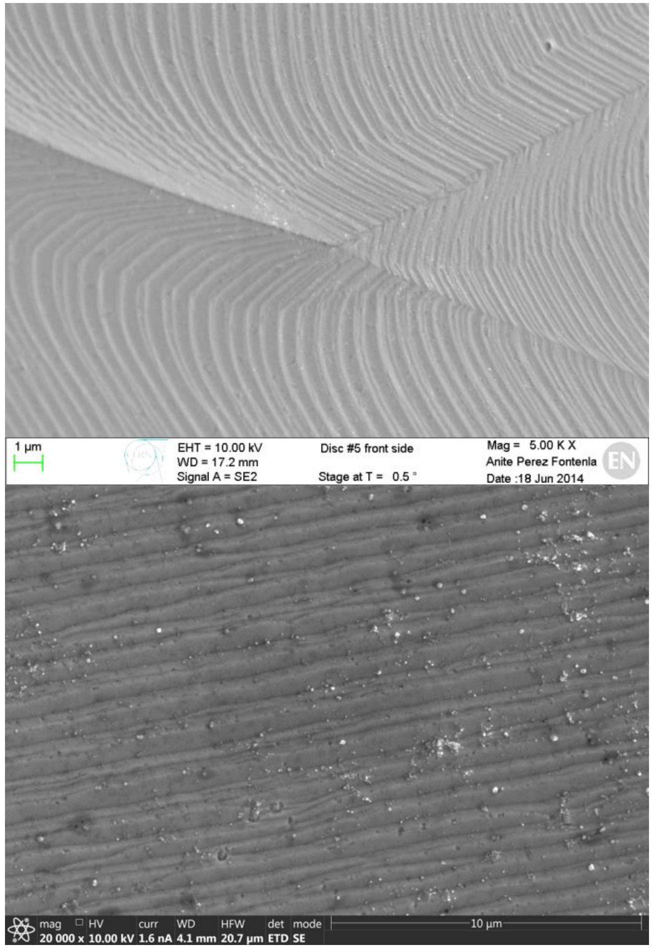
Editors' Suggestion

Theory of electric field breakdown nucleation due to mobile dislocations

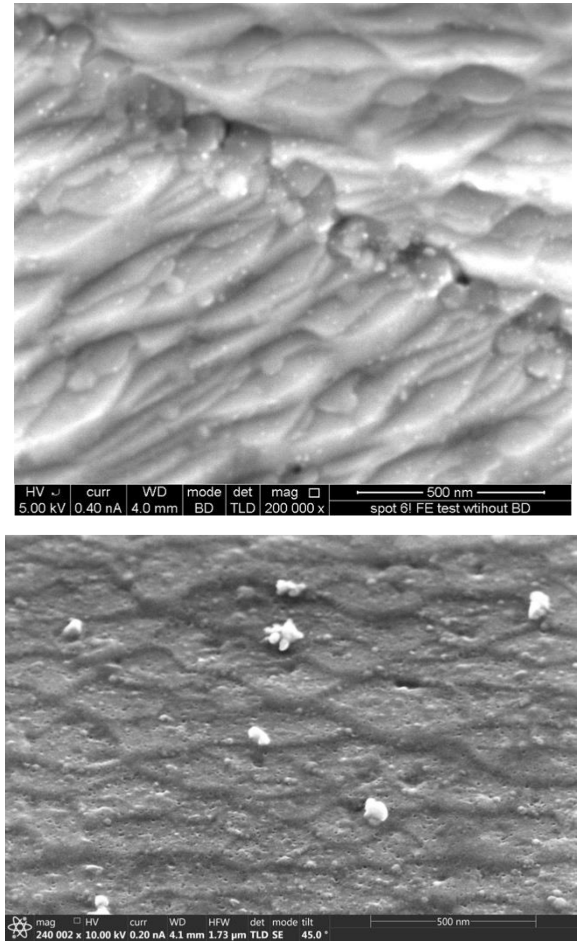
Pre BD –
dislocations below and observable on the surface.

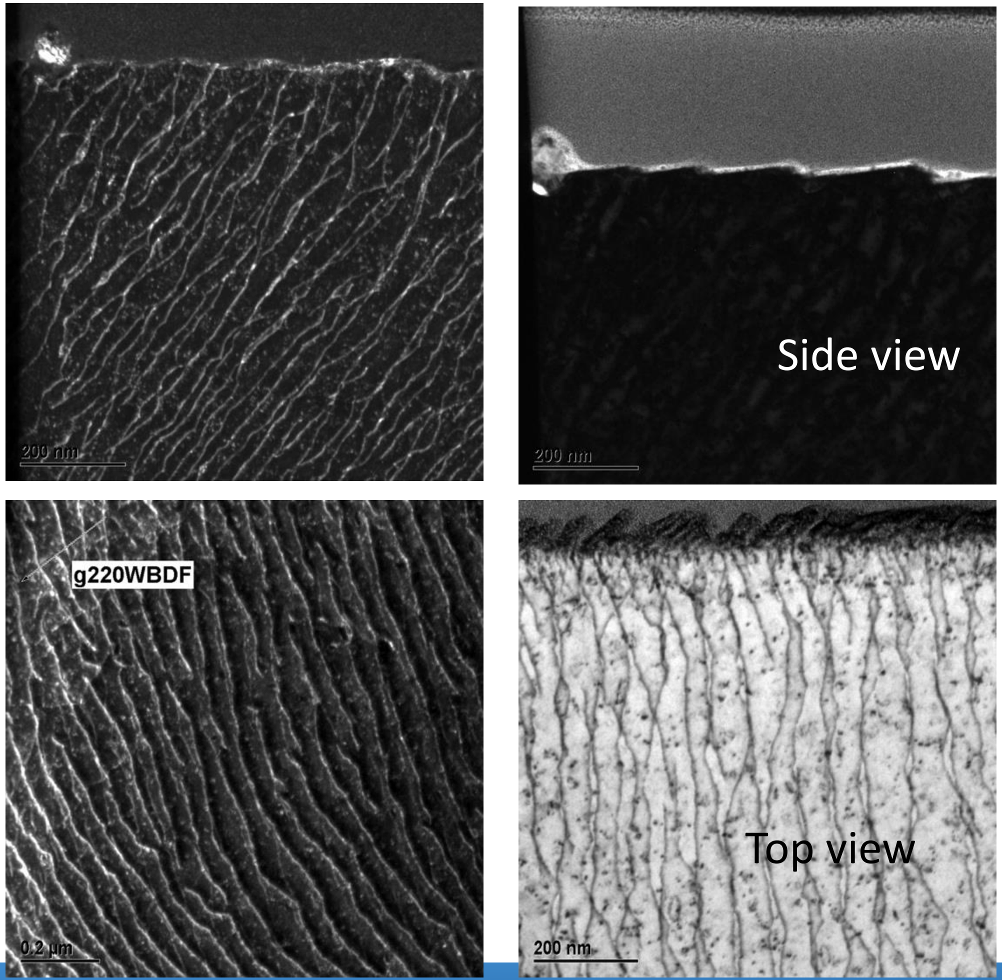
- periodic structures, sensitive to grain orientation
- These are manifestations of dislocation arrays

Terraces



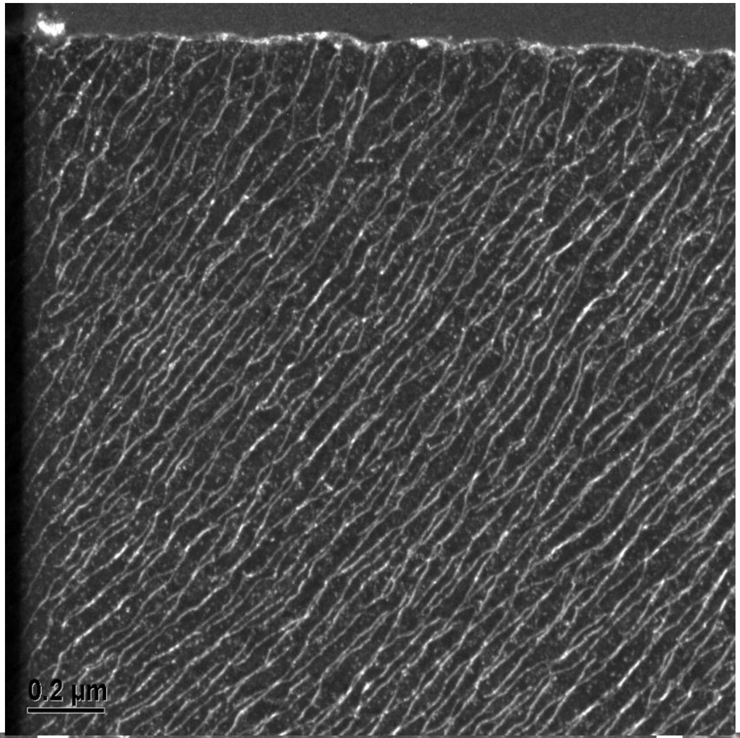
Fish Scales

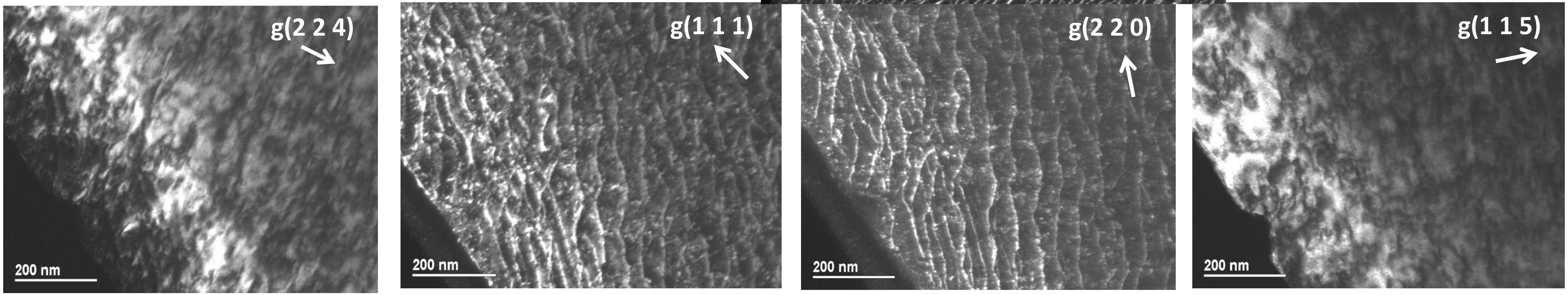




Quantifying dislocations properties

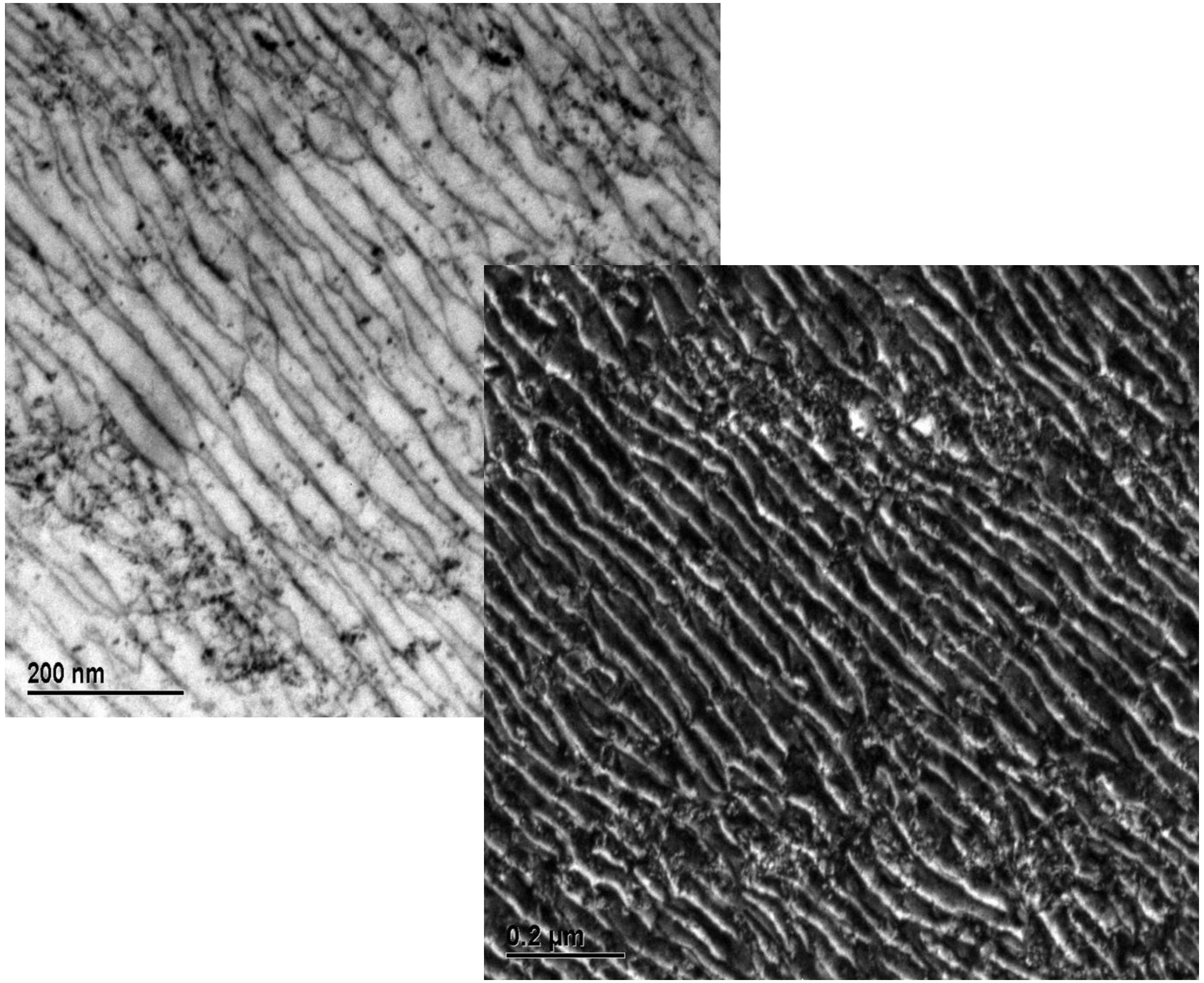
- Large grains with uniform dislocations patterns
- Normal density, but extremely coherent.
- Using various two-beam conditions dislocations identified as b=[110]
- Expected Edge and screw components for a dissociated mobile dislocation



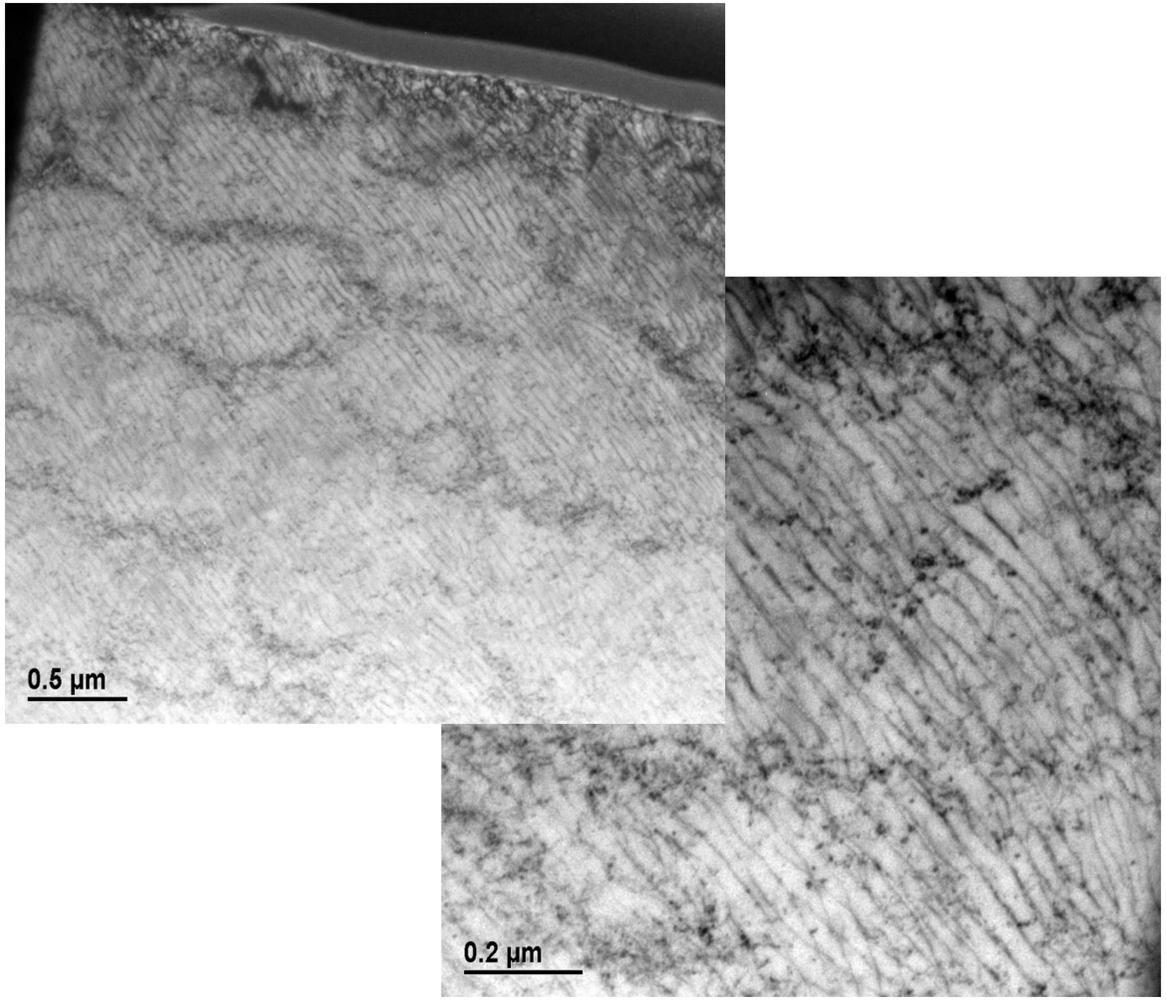


More details on dislocations characteristics

- Edges and constrictions

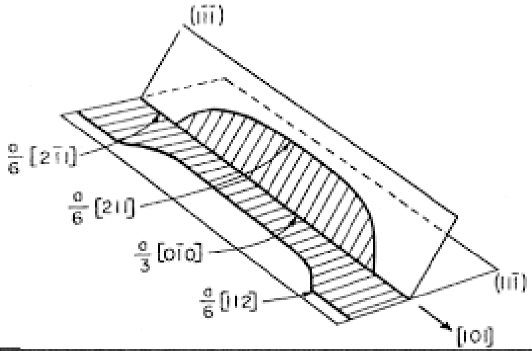


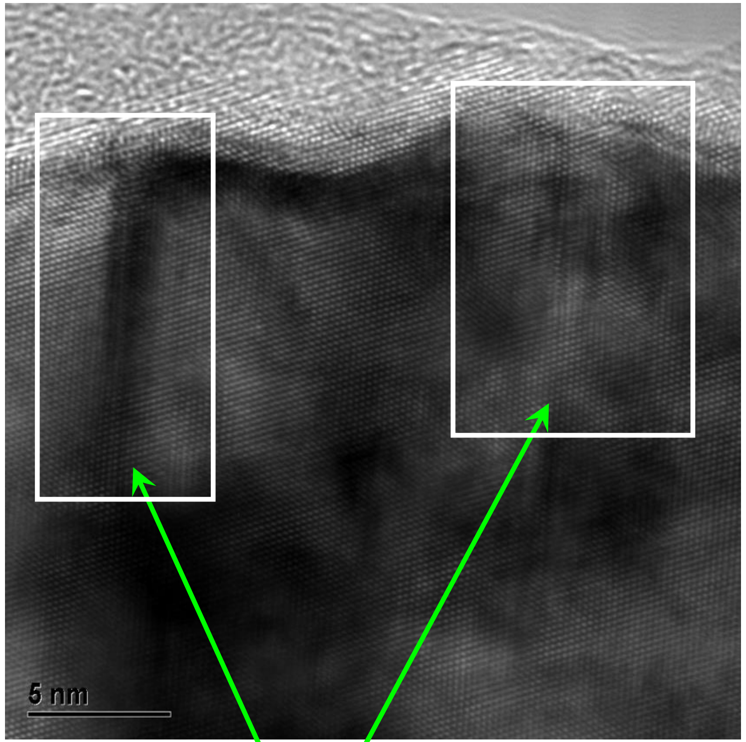
- Cellular structures

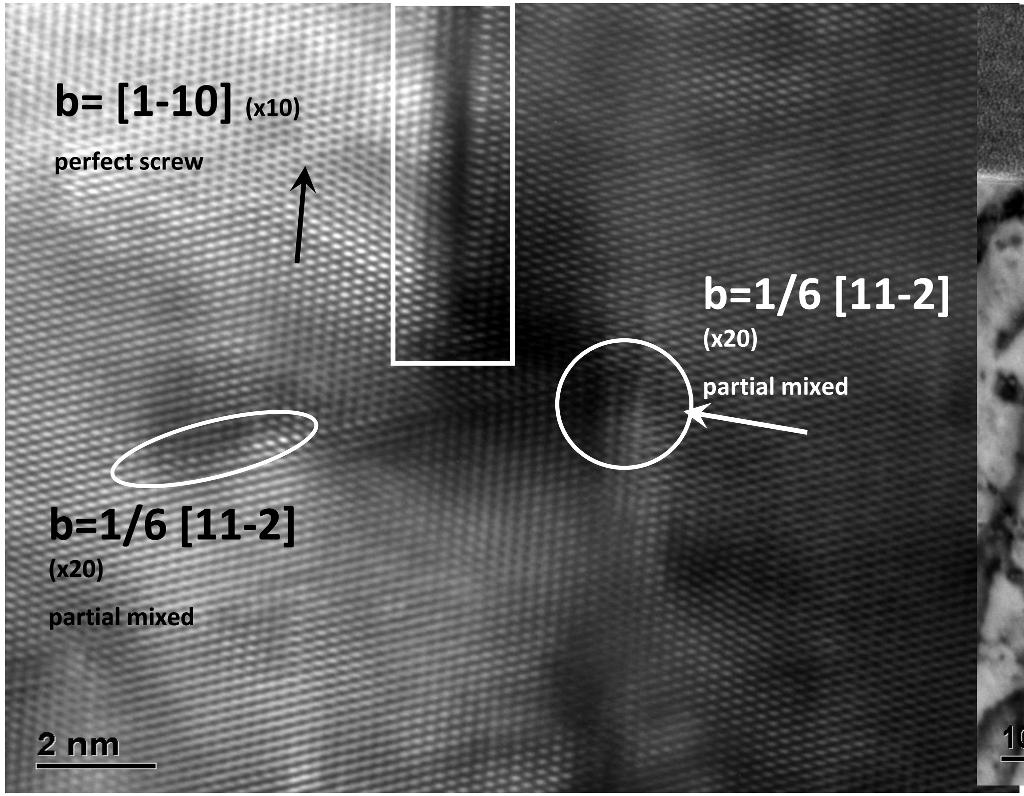


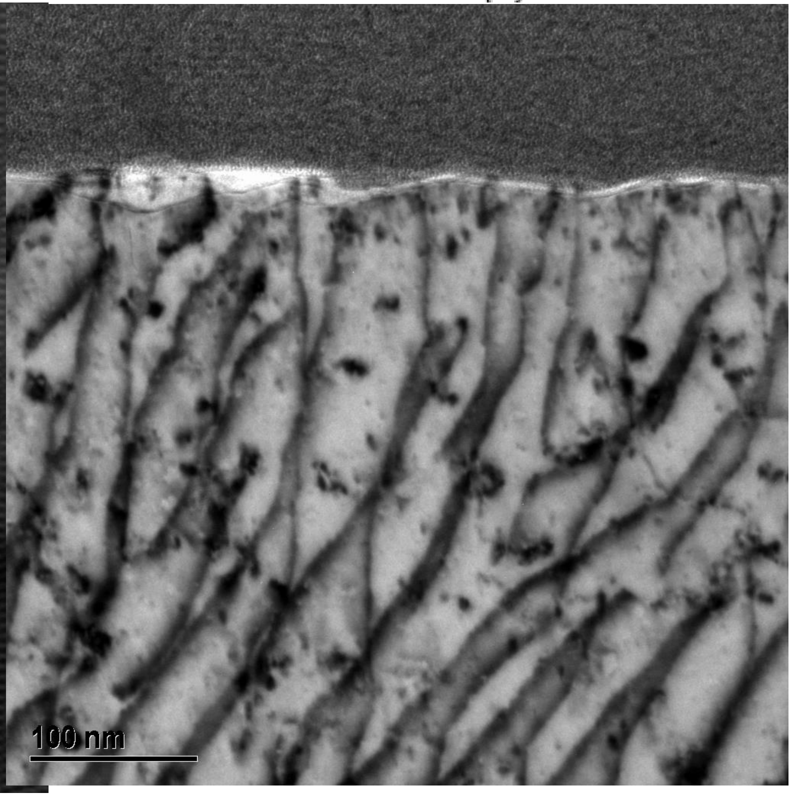
High res TEM -

- Expected mixed dislocations and stacking faults









Two identical groups of defects released to the surface to form terraces

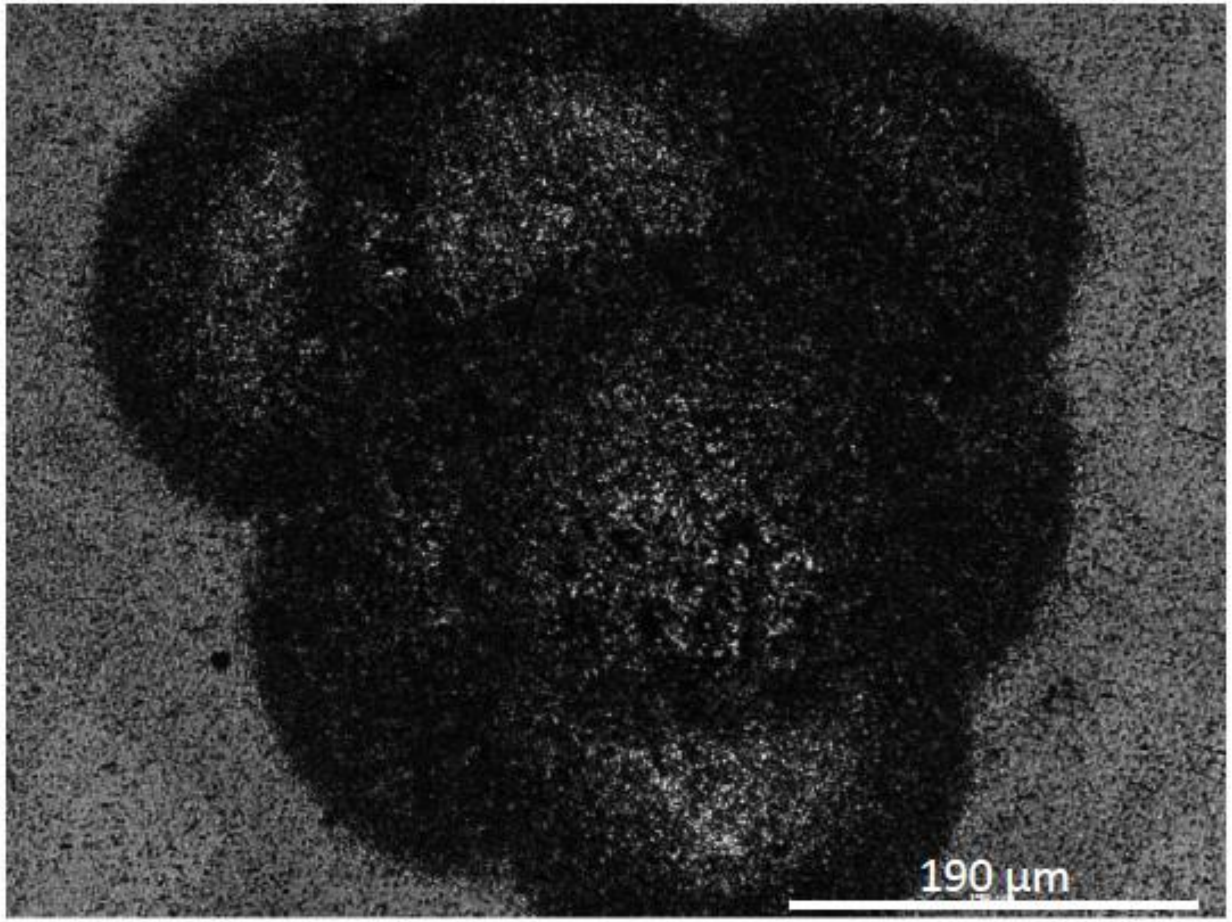
Conditioned surfaces –
do we see hardening – conditioning correlation?

- Dislocations based mechanism –
- Can we show that conditioning is related to hardness?

    (simple to measure ex-situ)

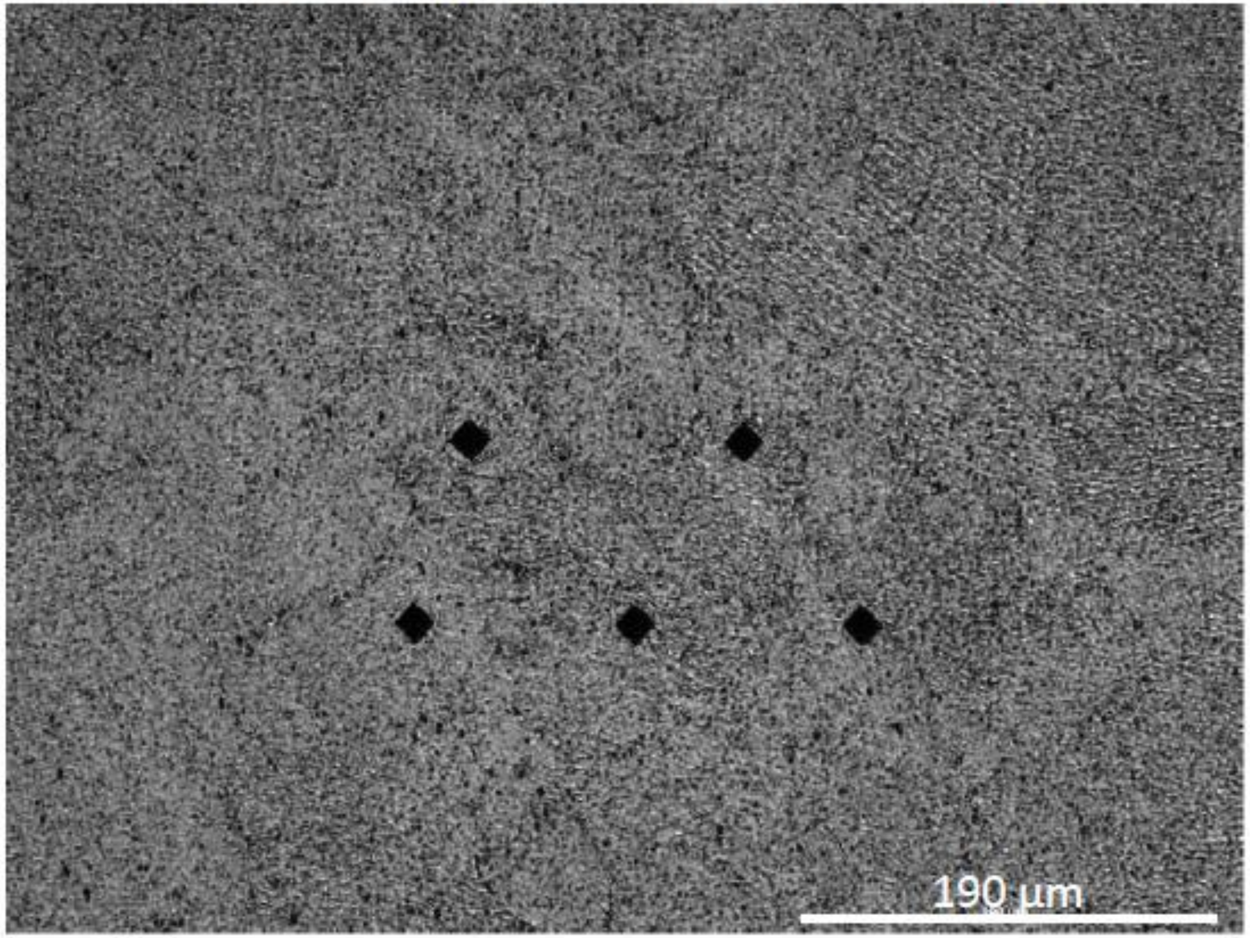
- Proving/Disproving – conditioning due to hardening…

Hard Cu, on a BD crater, at the center of the sample



Average hardness=(97±6)HV

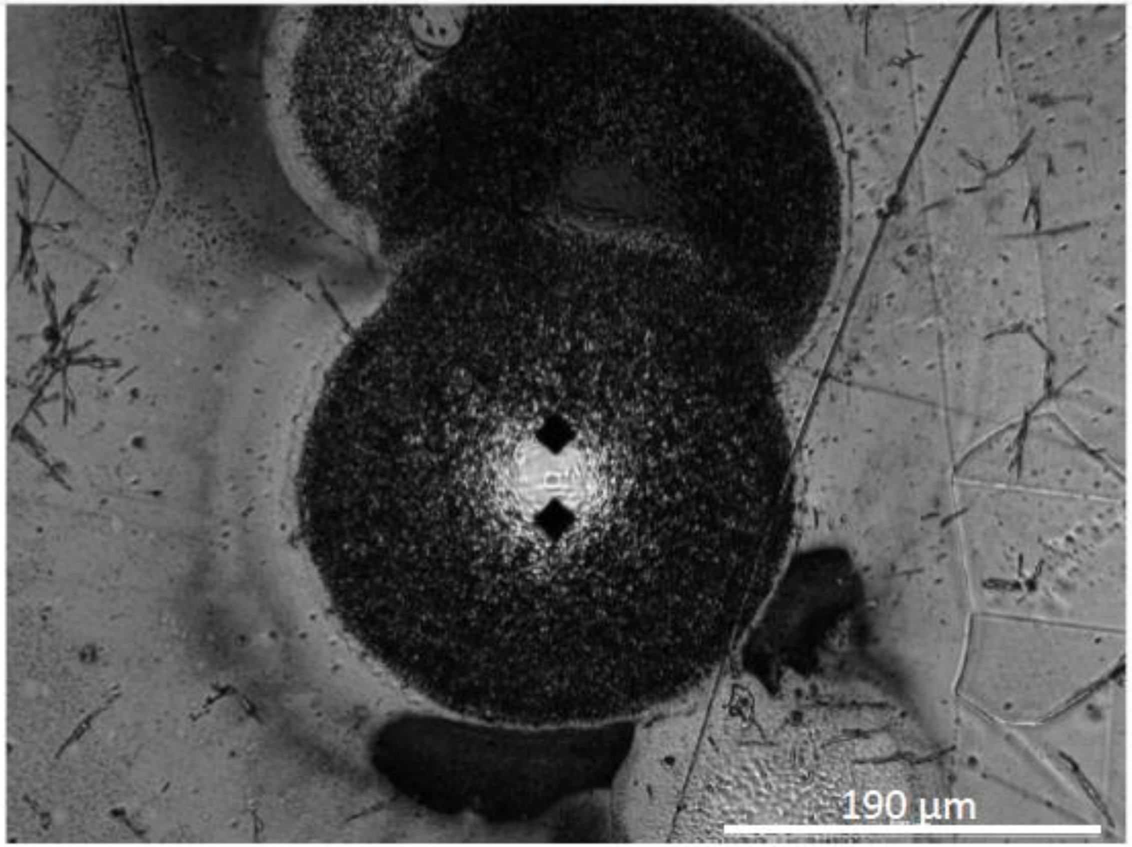
Hard Cu, a clean region in-between BD craters, at the center of the sample



Average hardness=(107.7±3.5)HV

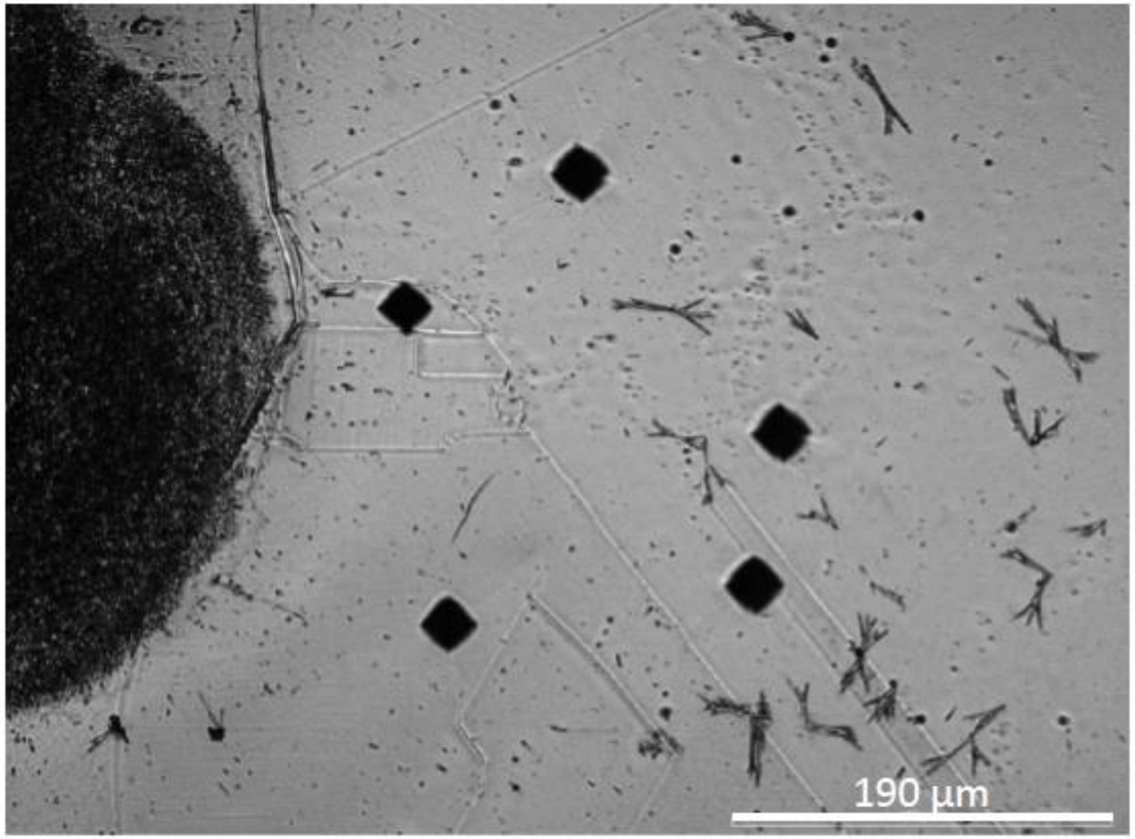
Magnification X100, a load of 20grf, loading time 10s

Soft Cu, , on a BD crater, at the center of the sample



Average hardness=(76.5±7.1)HV

Soft Cu, a clean region in-between BD craters at the center of the sample

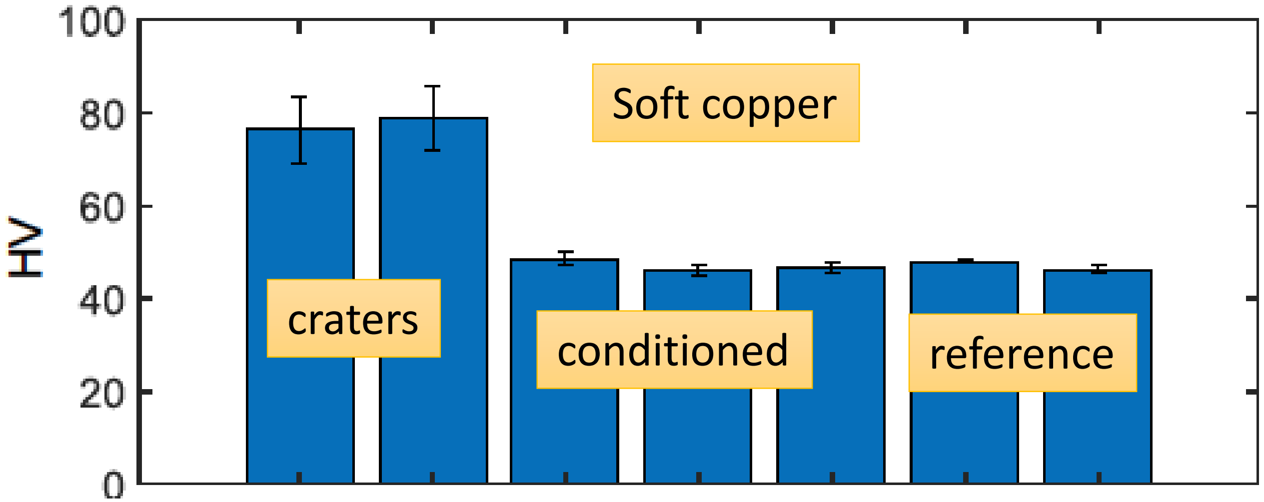


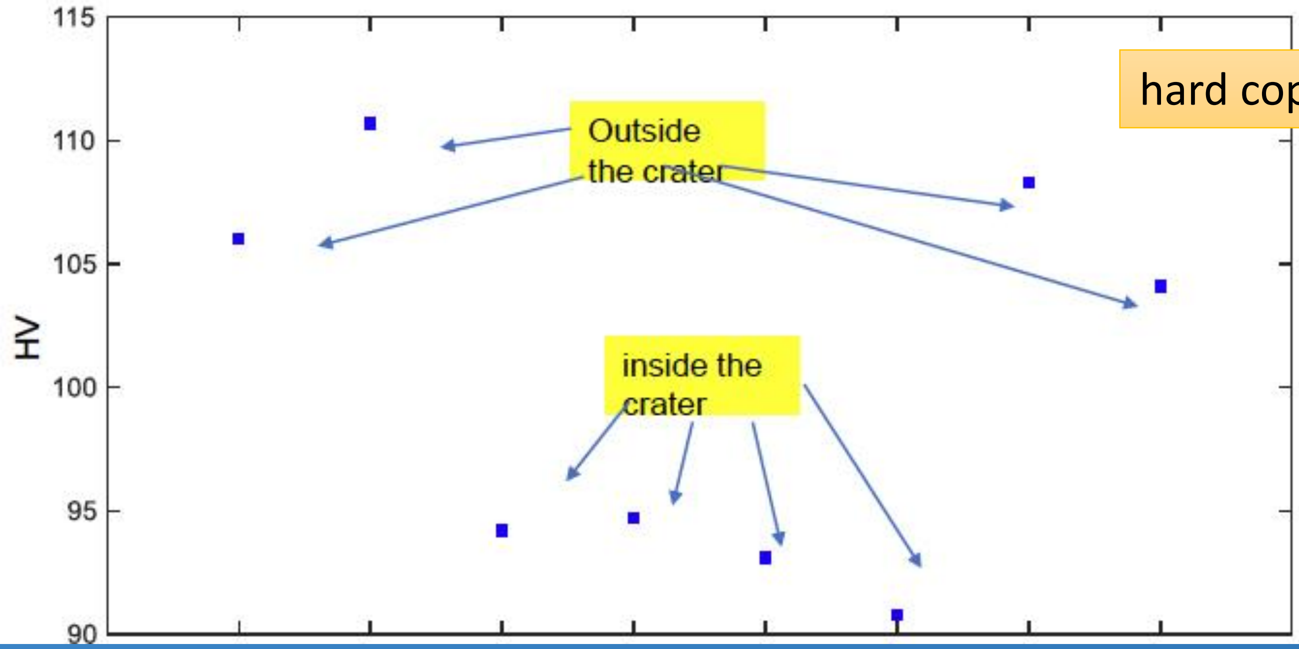
Average hardness=(48.6±1.5)HV

Magnification X100, a load of 20grf, loading time 10s

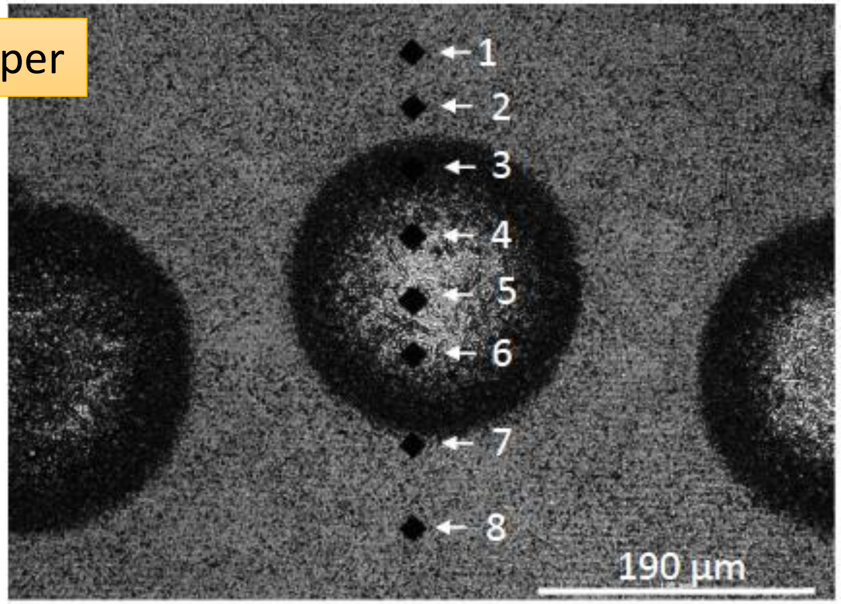
Hardness modifications at craters..

- No clear hardness – conditioning correlation
- Softer regions in hard Cu
  - would have lead to repeat BD.
  - Should have seen a strong difference in bd locations correlation

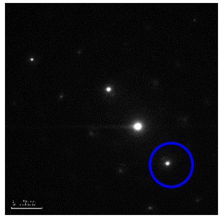




hard copper

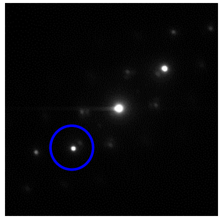


Not so significant density modifications



Reference

RF 23



Observable density is similar in
Various orientations:
Cross-section versus Top view

And is not affected by sample thickness (100-200 nm)
(one system is observable)

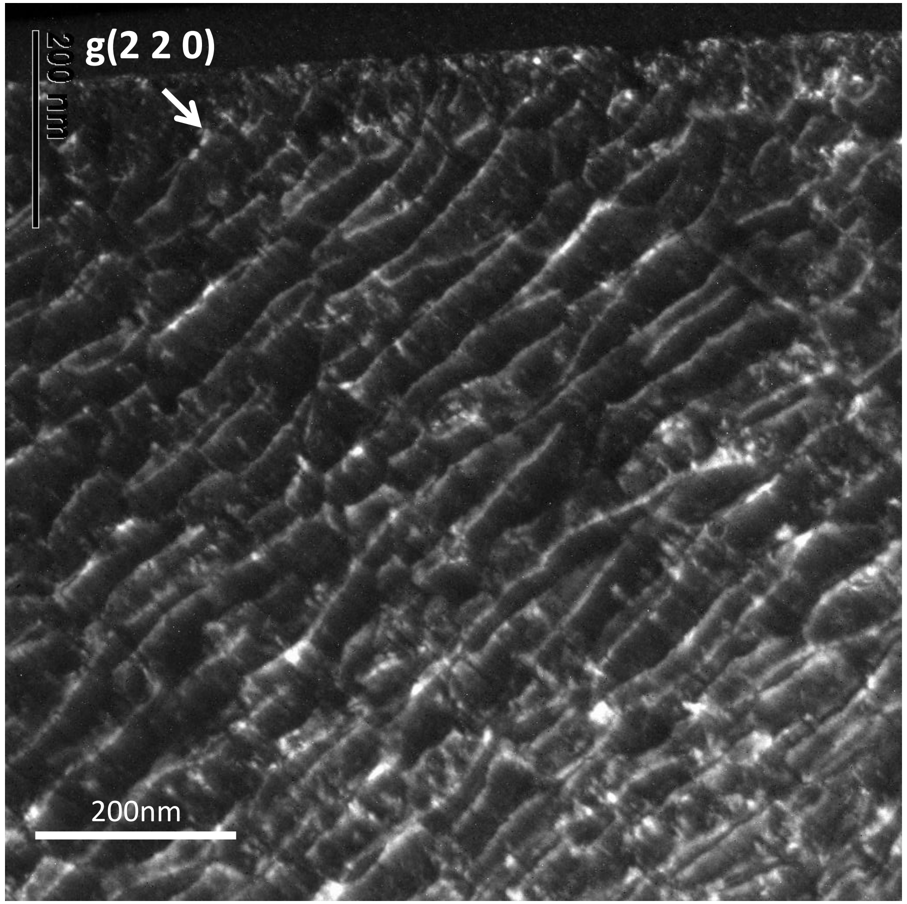
Rf exposed:

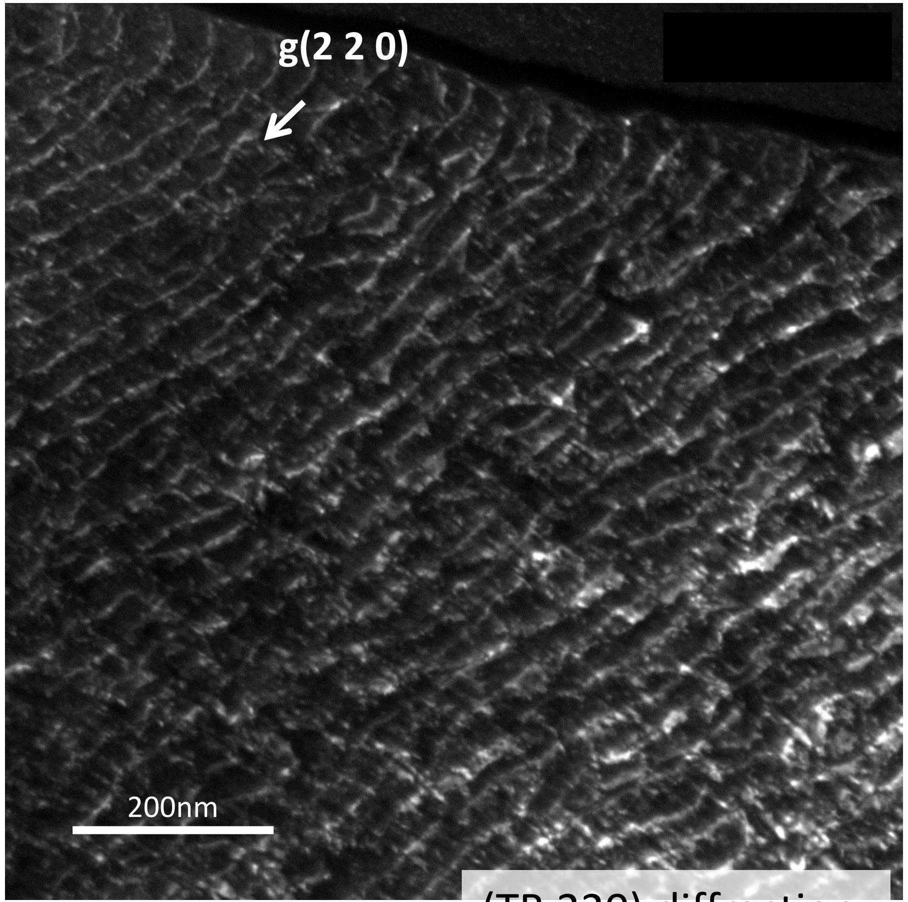
$$\rho_s = 4 \pm 0.2\ (10^5 cm^{-1})$$

Pristine:

$$\rho_s = 3.1 \pm 0.5\ (10^5 cm^{-1})$$

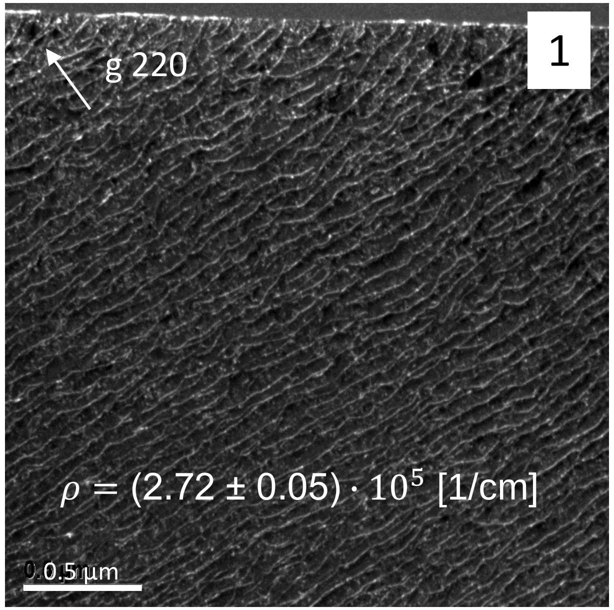
Not consistent….



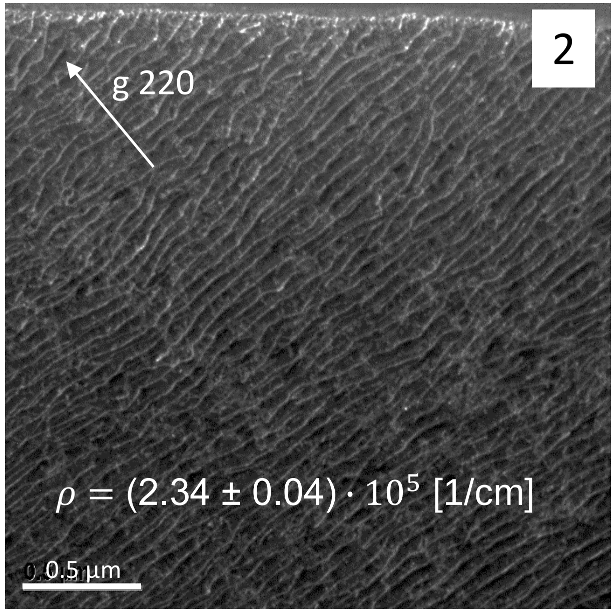


(TB 220) diffraction

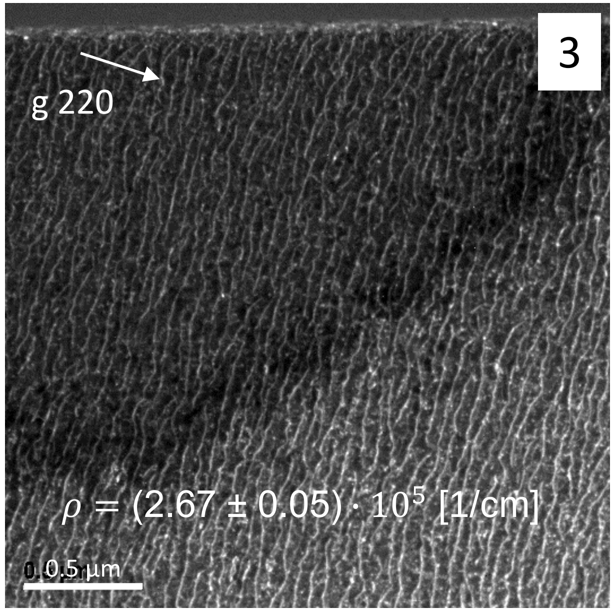
Reference



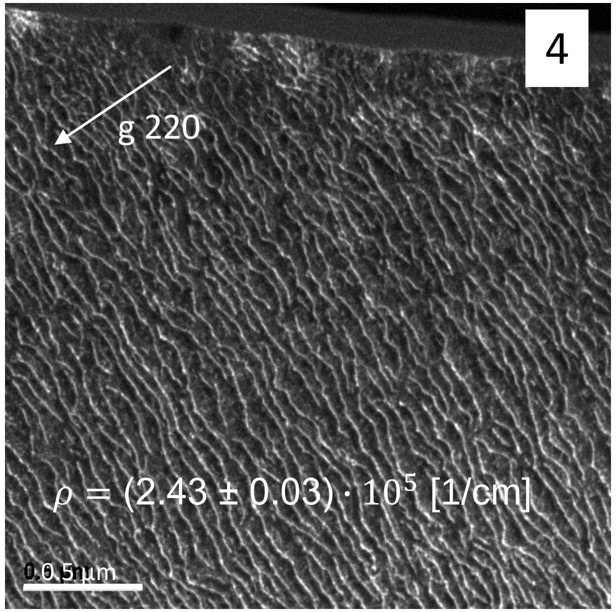
RF (Crab-cavity sample)



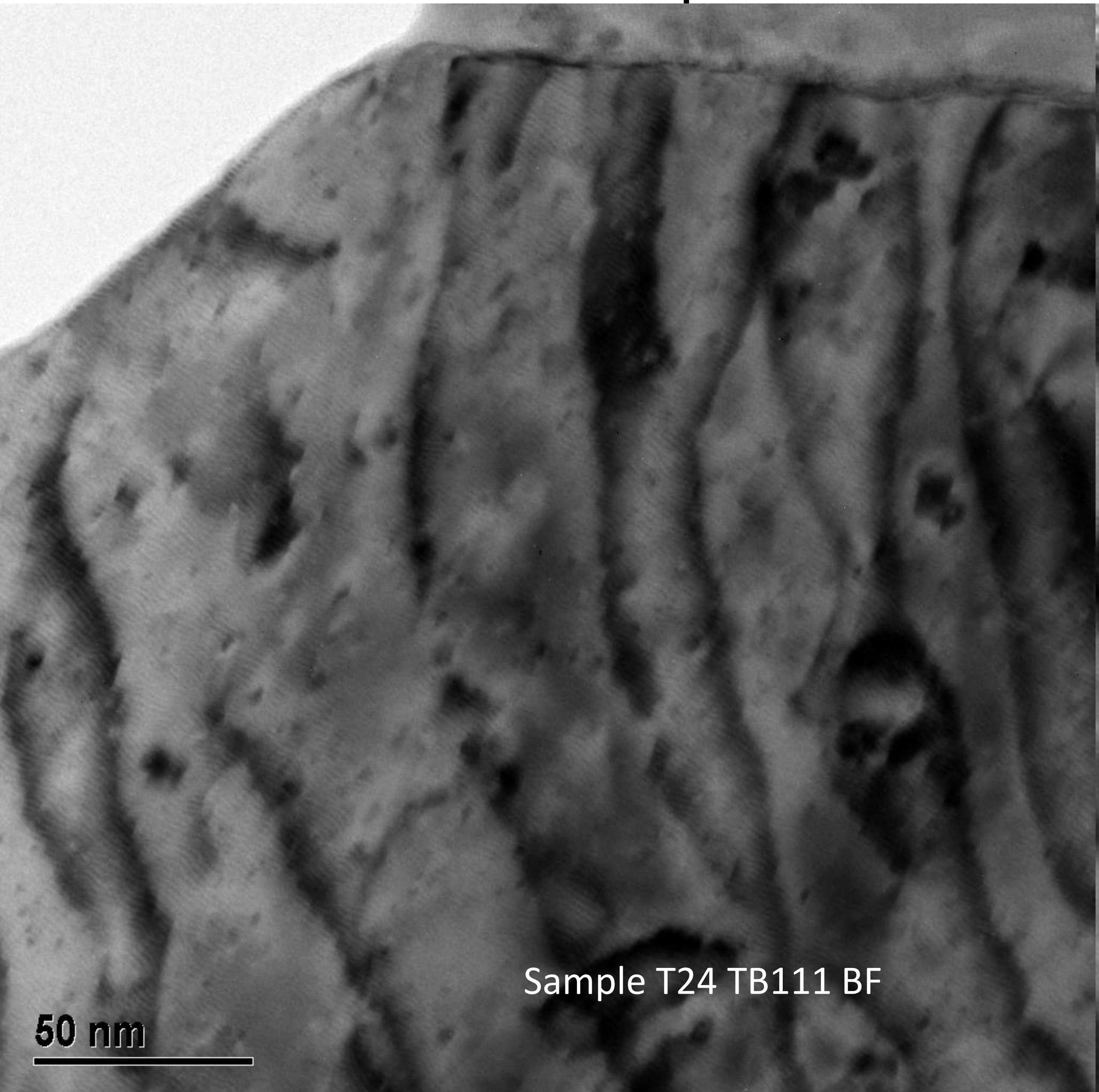
FA half conditioned

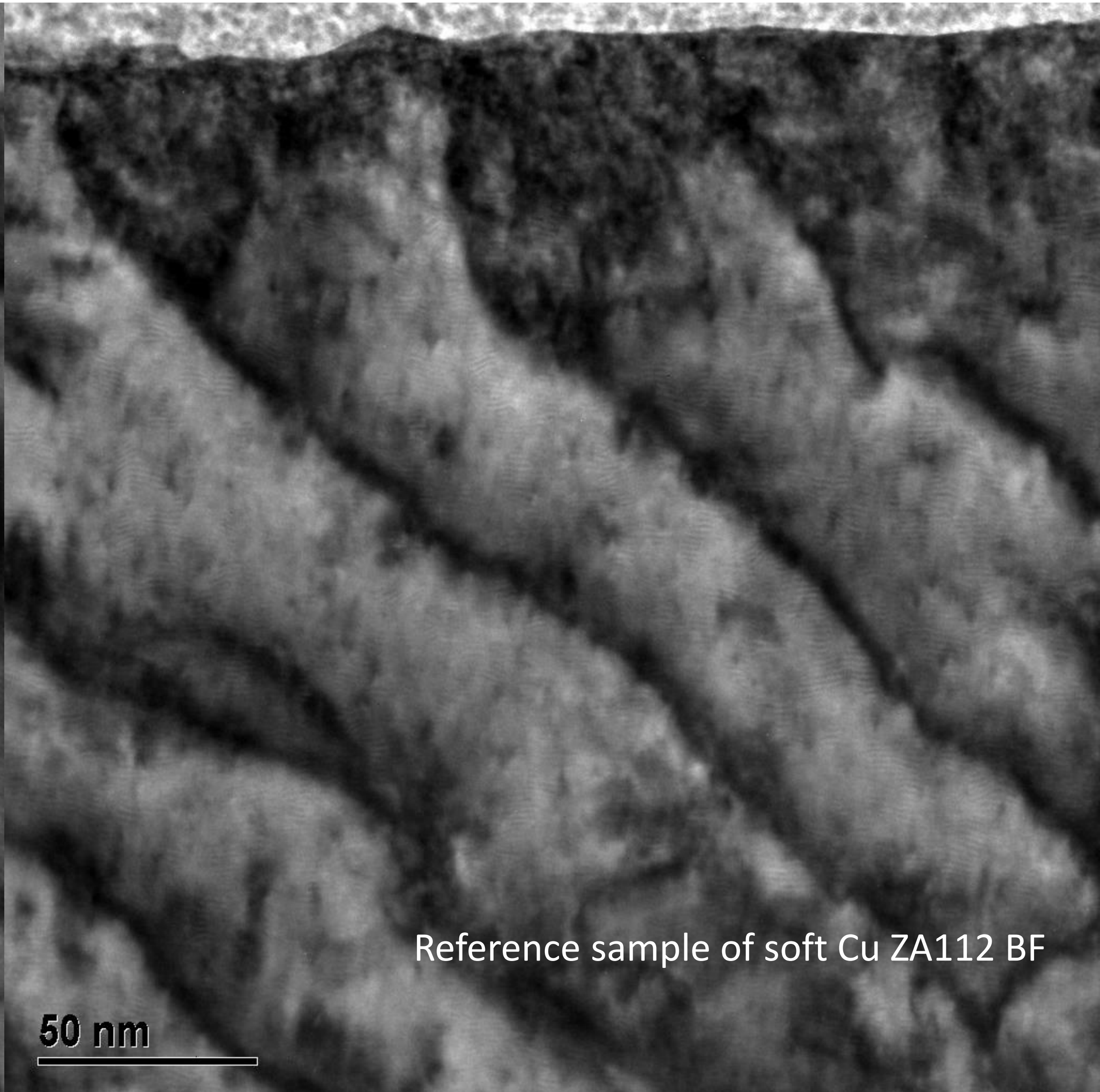


FA full conditioned

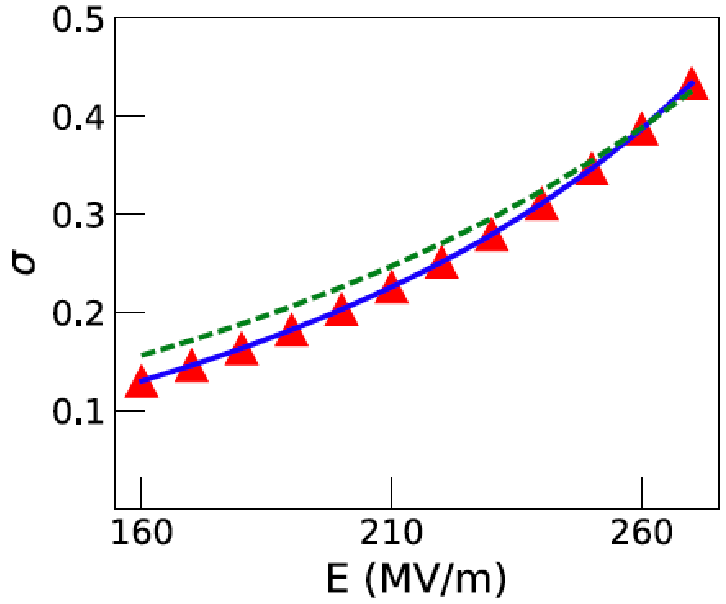


Zoom in on top 100 nm





- Dislocations are stabilized in an ordered array.
- Model based on critical fluctuations in mobile dislocations, is consistent with observables
- But - no clear dislocations based conditioning…
- We skip the missing link (dislocations – BD nucleations) and try to identify pre-BD fluctuations…
  - Dark current (common to most models)
  - Acoustic emission – dislocations specific



PRE-BD signals

- As the system approaches the critical point. Fluctuation diverge.

- Observable through standard deviation of the time correlation

$$SD(t) = \frac{\int_{t-\Delta}^{t+\Delta} \left(I(t) - <I>\right)^2 dt}{\left(<I>\right)^2}$$

- Or, more generally, autocorrelation in the signal

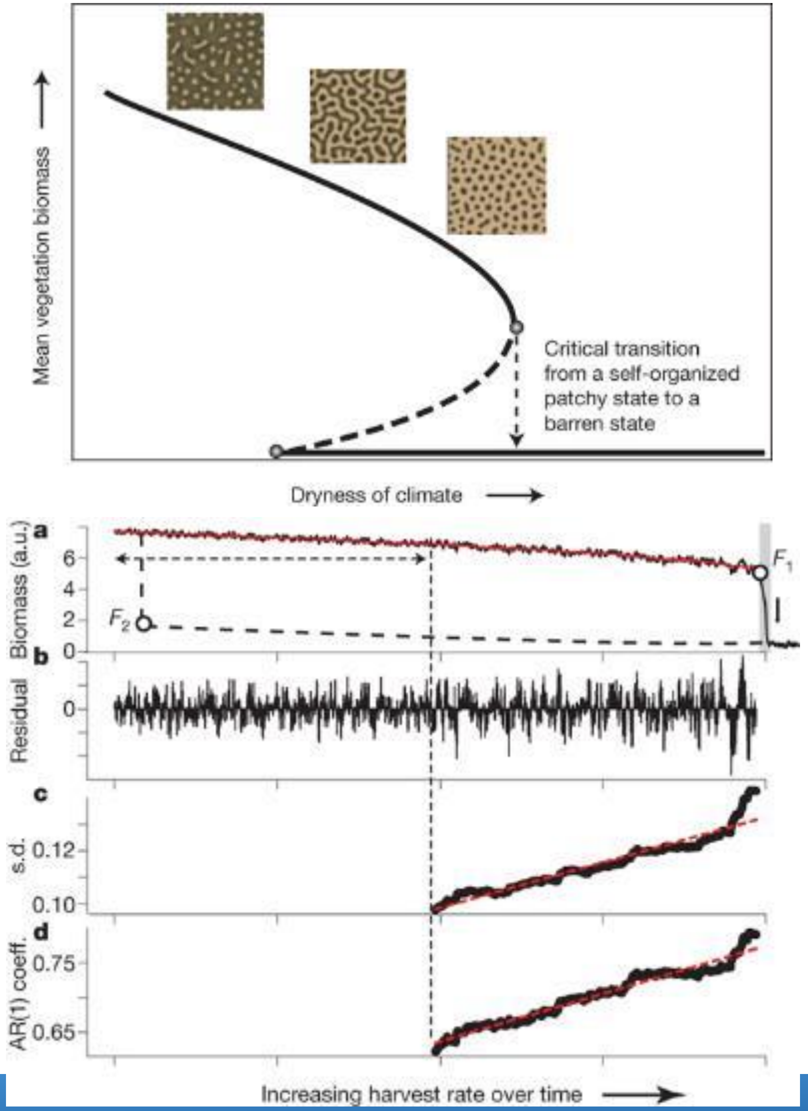
$$R(k) = \frac{\int_0^{t-k} \left(I(t) - <I>\right)\left(I(t+k) - <I>\right) dt}{\int_0^{t-k} \left(I(t) - <I>\right)^2 dt}$$

Vol 461|3 September 2009|doi:10.1038/nature08227 nature

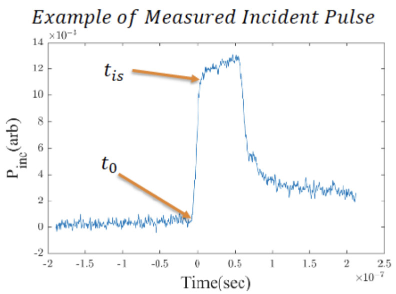
REVIEWS

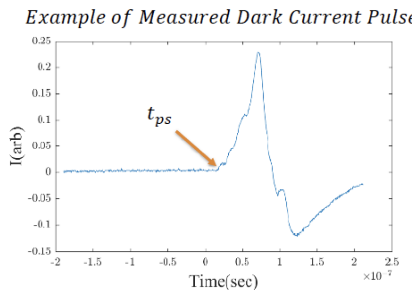
Early-warning signals for critical transitions

Marten Scheffer[1], Jordi Bascompte[2], William A. Brock[3], Victor Brovkin[5], Stephen R. Carpenter[4], Vasilis Dakos[1], Hermann Held[6], Egbert H. van Nes[1], Max Rietkerk[7] & George Sugihara[8]



Fluctuation Analysis in dark currents
(Jan Paszkiewicz, Sagy Lachman and Iaroslava Profatilova)

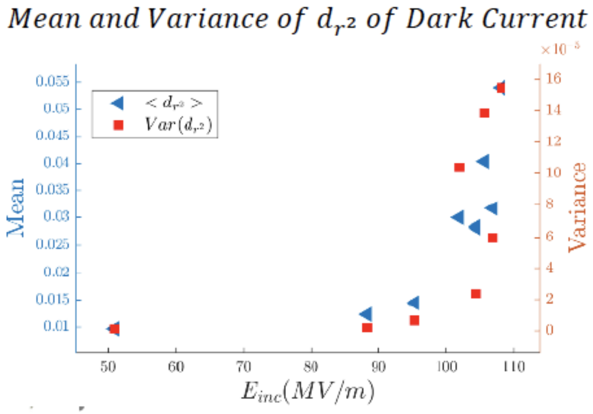


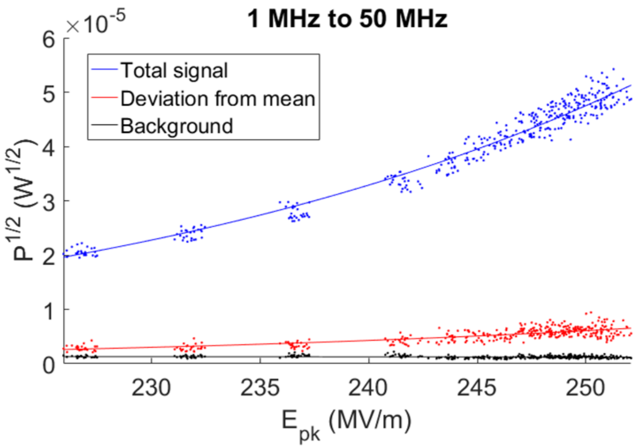


Identifying pre BD fluctuations is problematic…

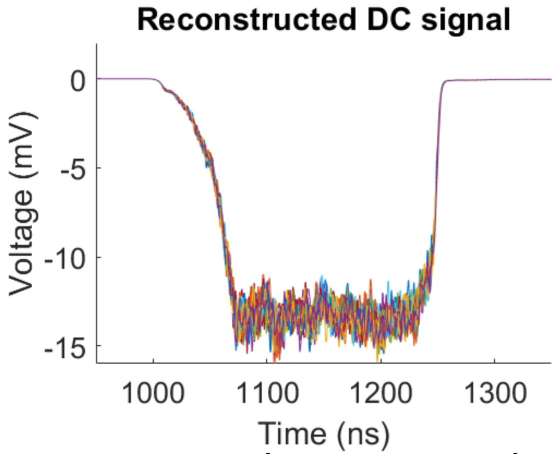
Fluctuations – need to establish a reference signal.
In RF – transfer function? Variation in applied field?
Must have a conditioned sample – (low field extrinsic BD do not count!)

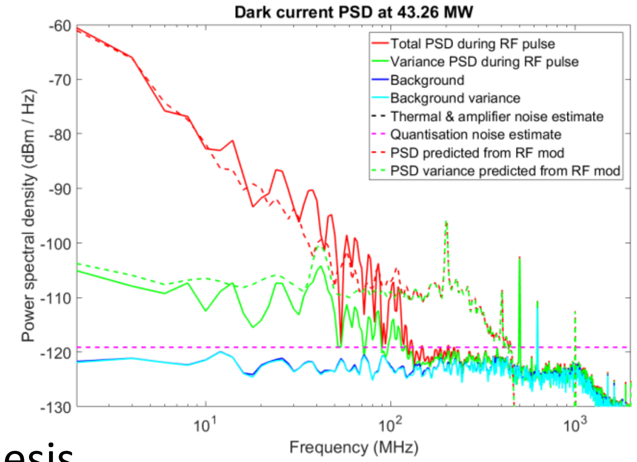
led to Sagy's work on fluctuations analysis of beta
(See Sagy's talk tomorrow)
And, to efforts using DC FGS system to identify fluctuations.
See Jan Paszkiewicz and Iarrosalva Profatilova poster.





Go to Jan's poster and see for yourself!



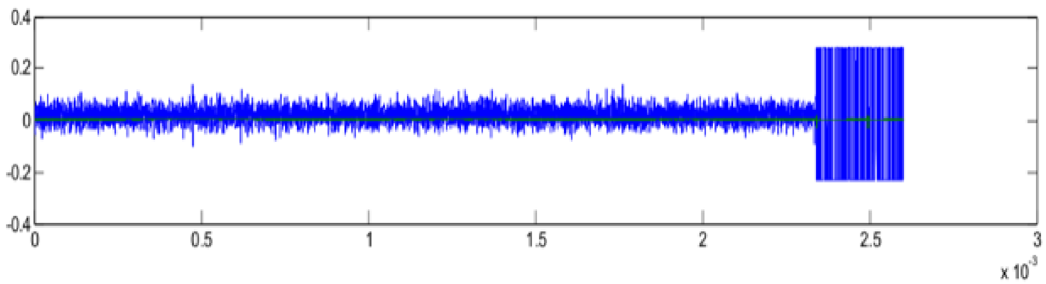


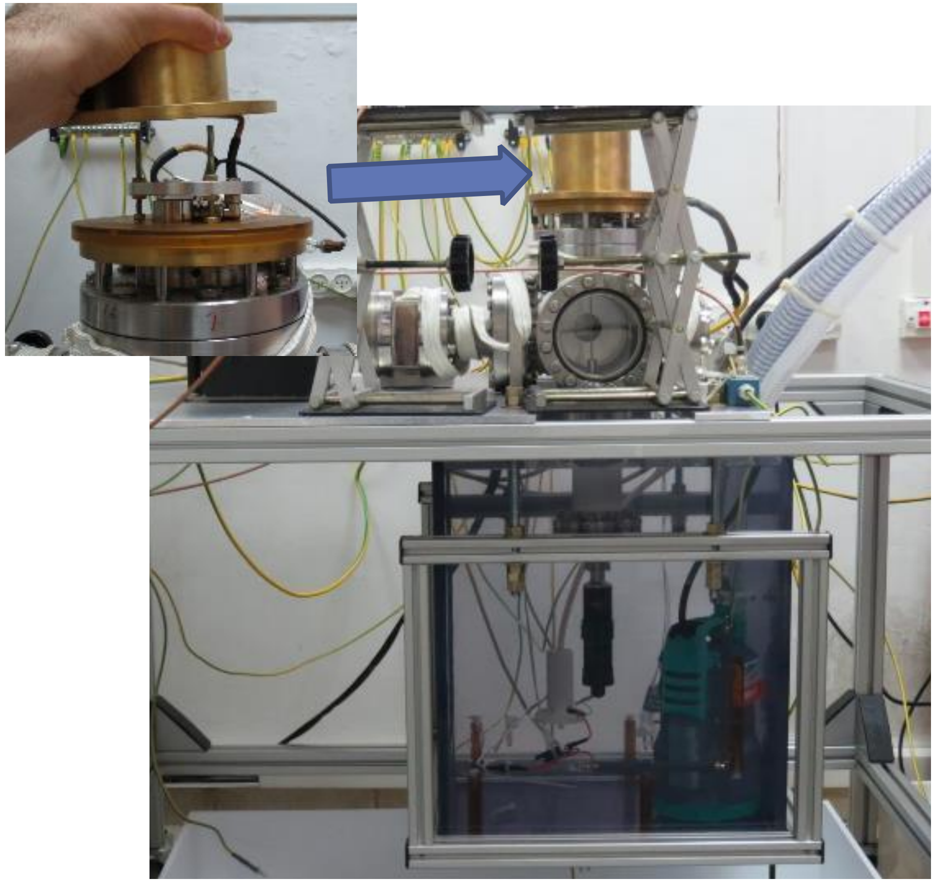
Details in Jan Paszkiewicz thesis….

Acoustic emission measurements

(under development – Itay Nachshon, Raanan Gad, Sagy Lachman)

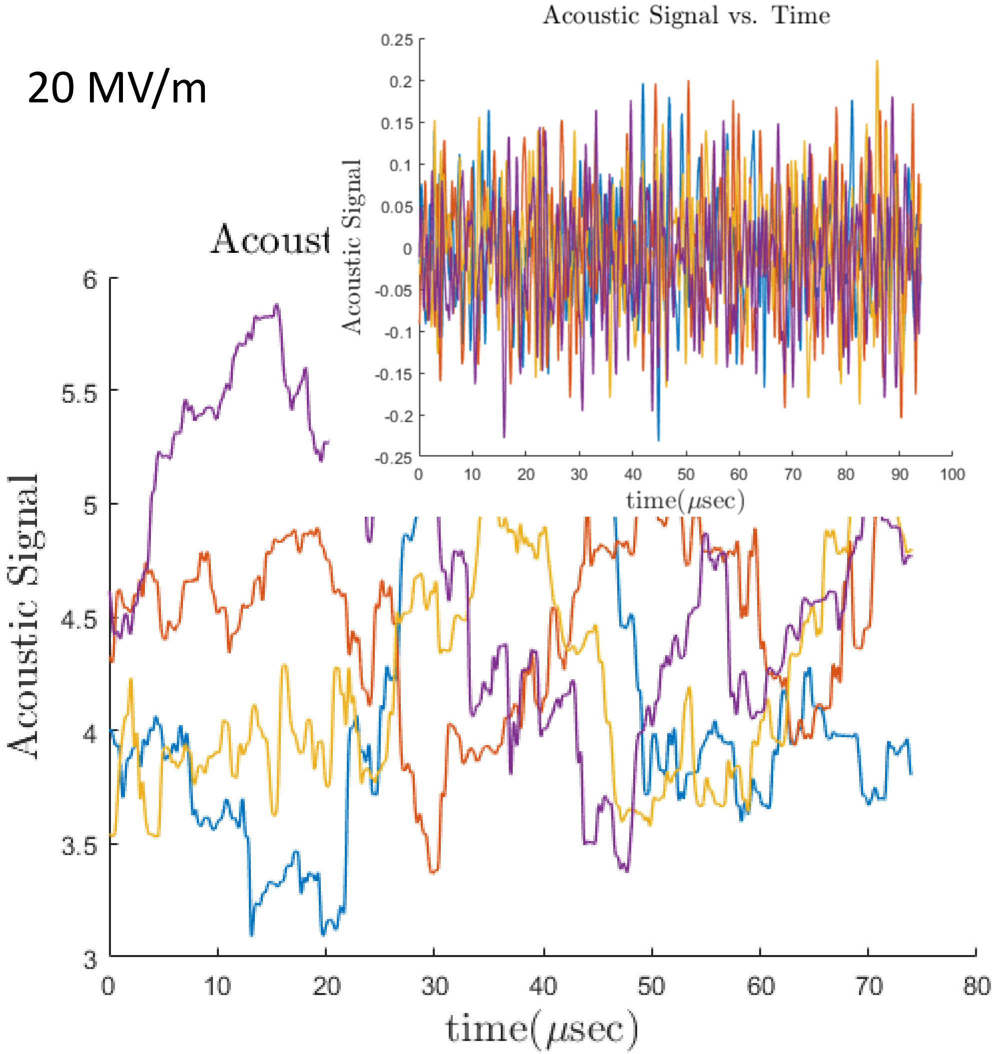
- Acoustic emission – distinctive signal from moving dislocations.
- System composed
- Questions:
  Can we identify pre BD fluctuations?
  Correlate current and AE signals?



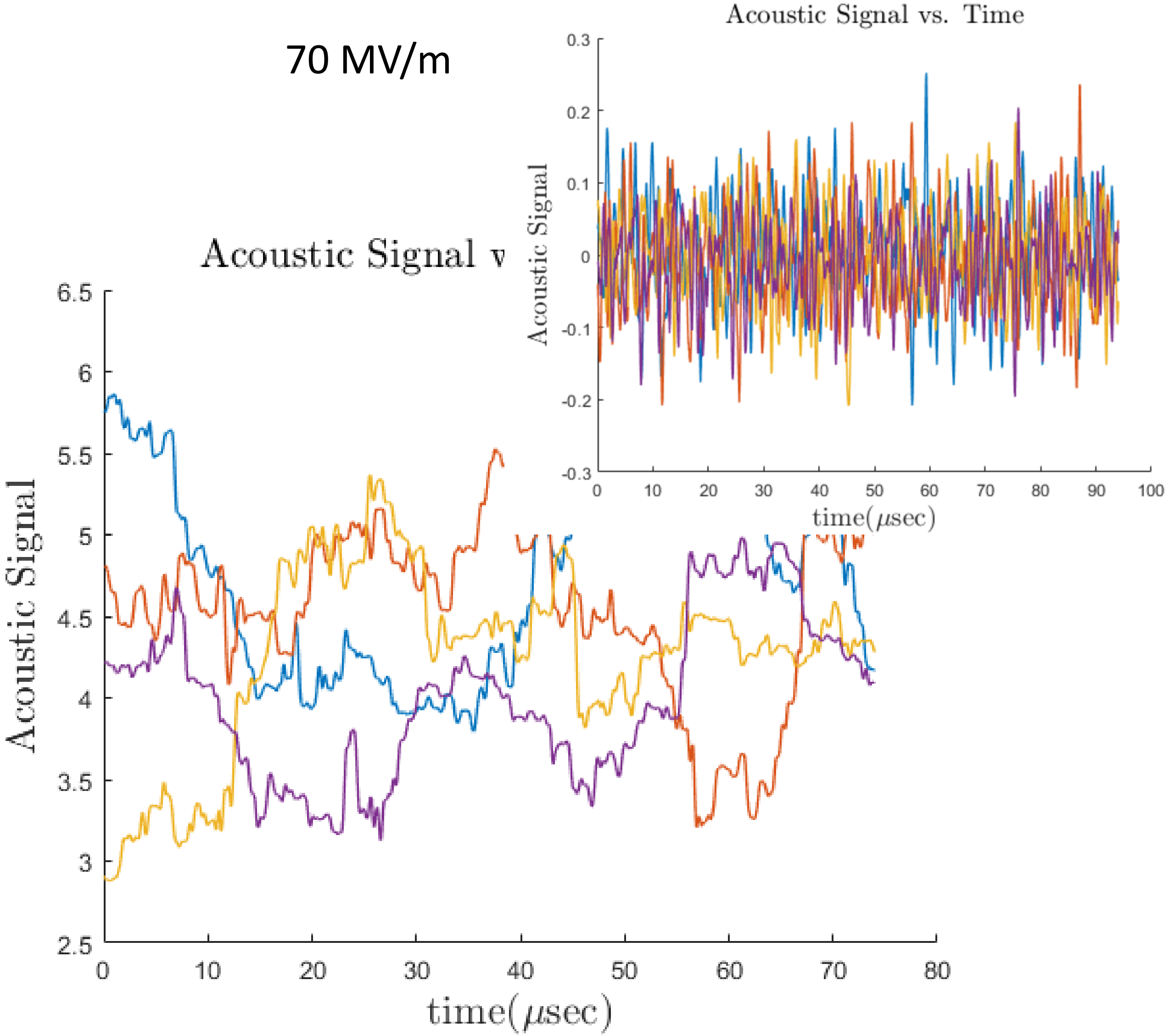


First trials – no real AE- increase with E

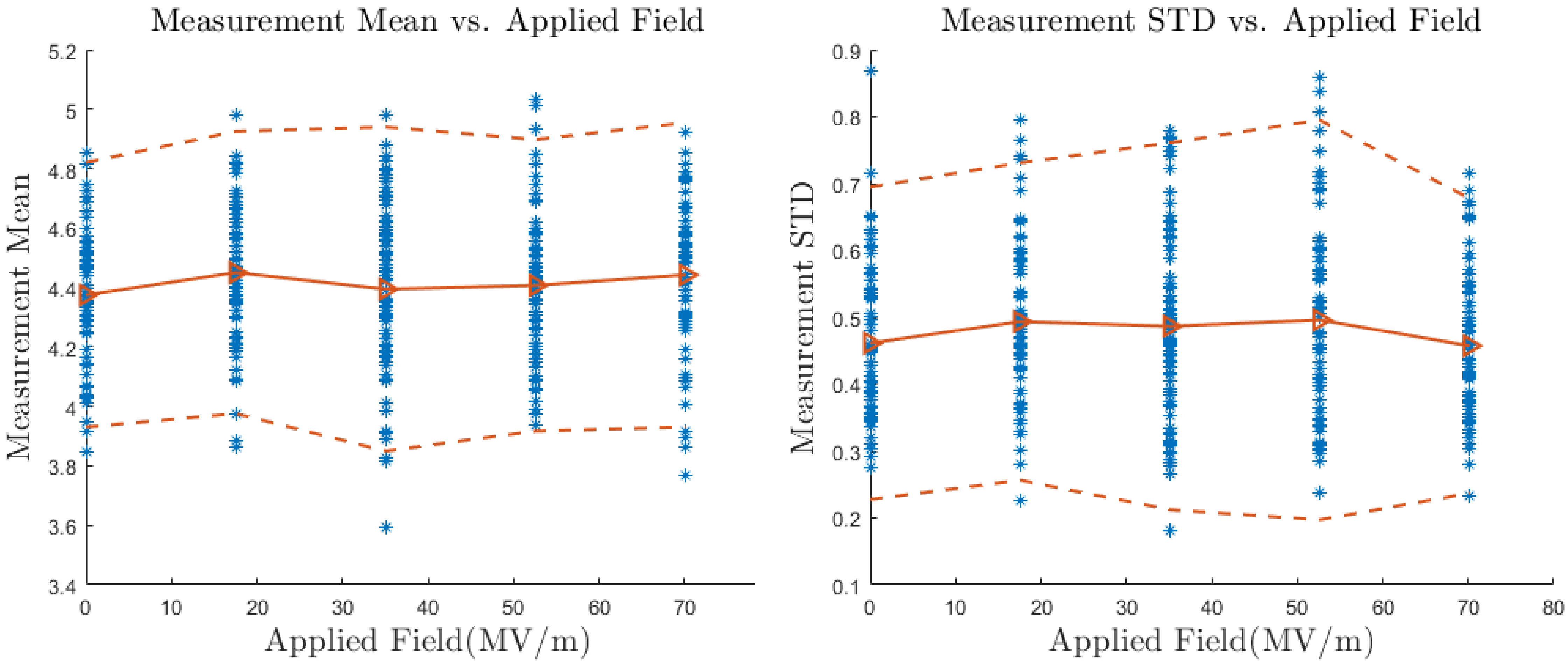
20 MV/m



70 MV/m

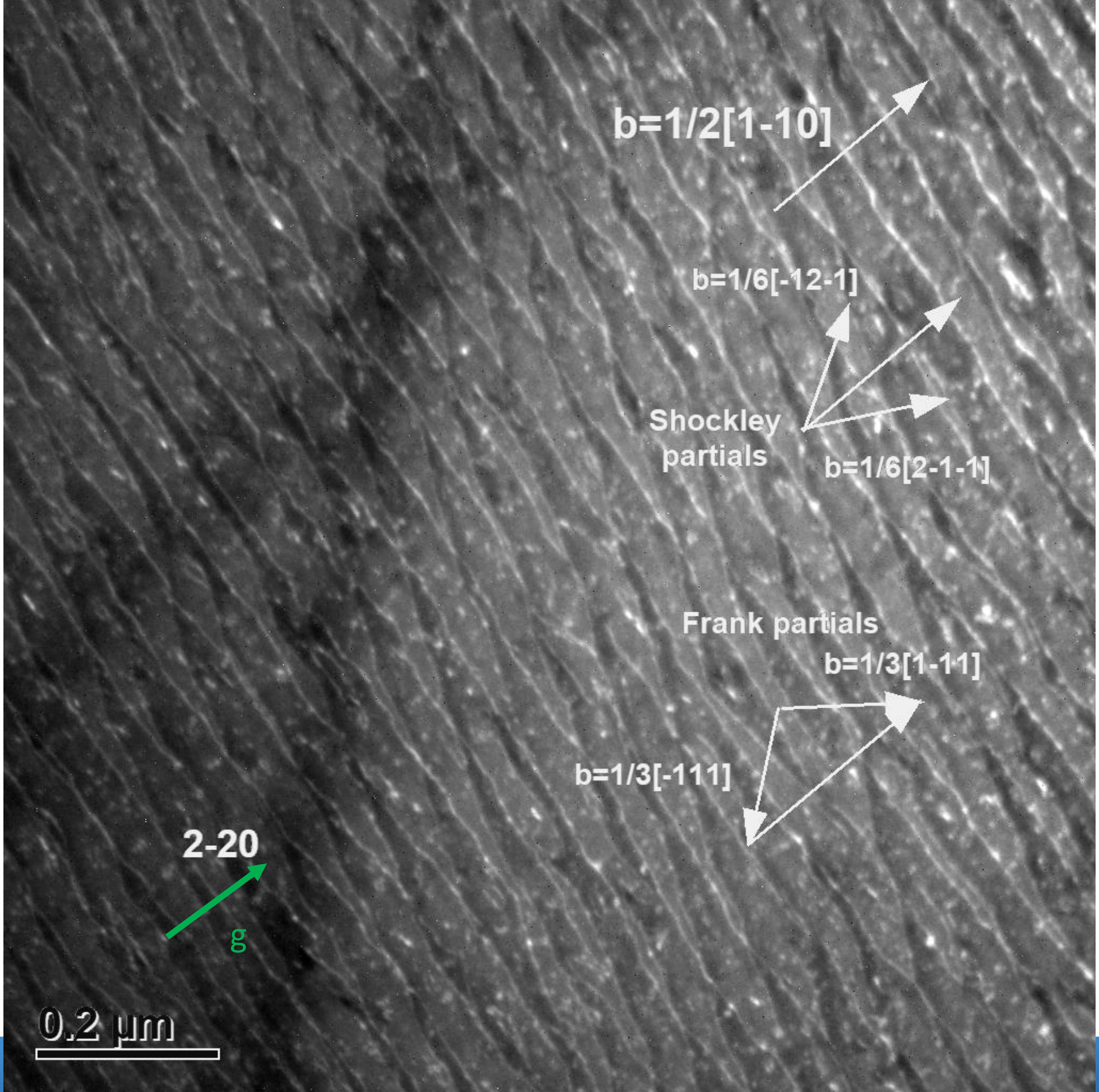


No real signal below BD

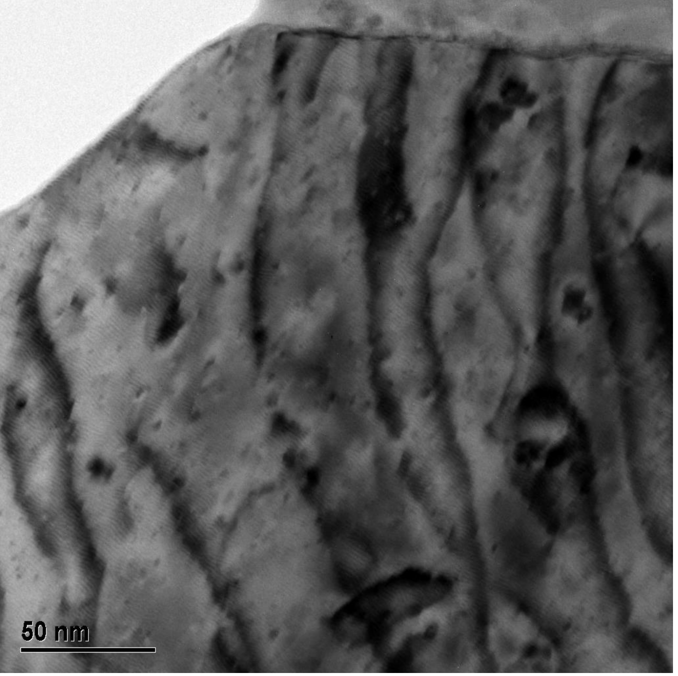


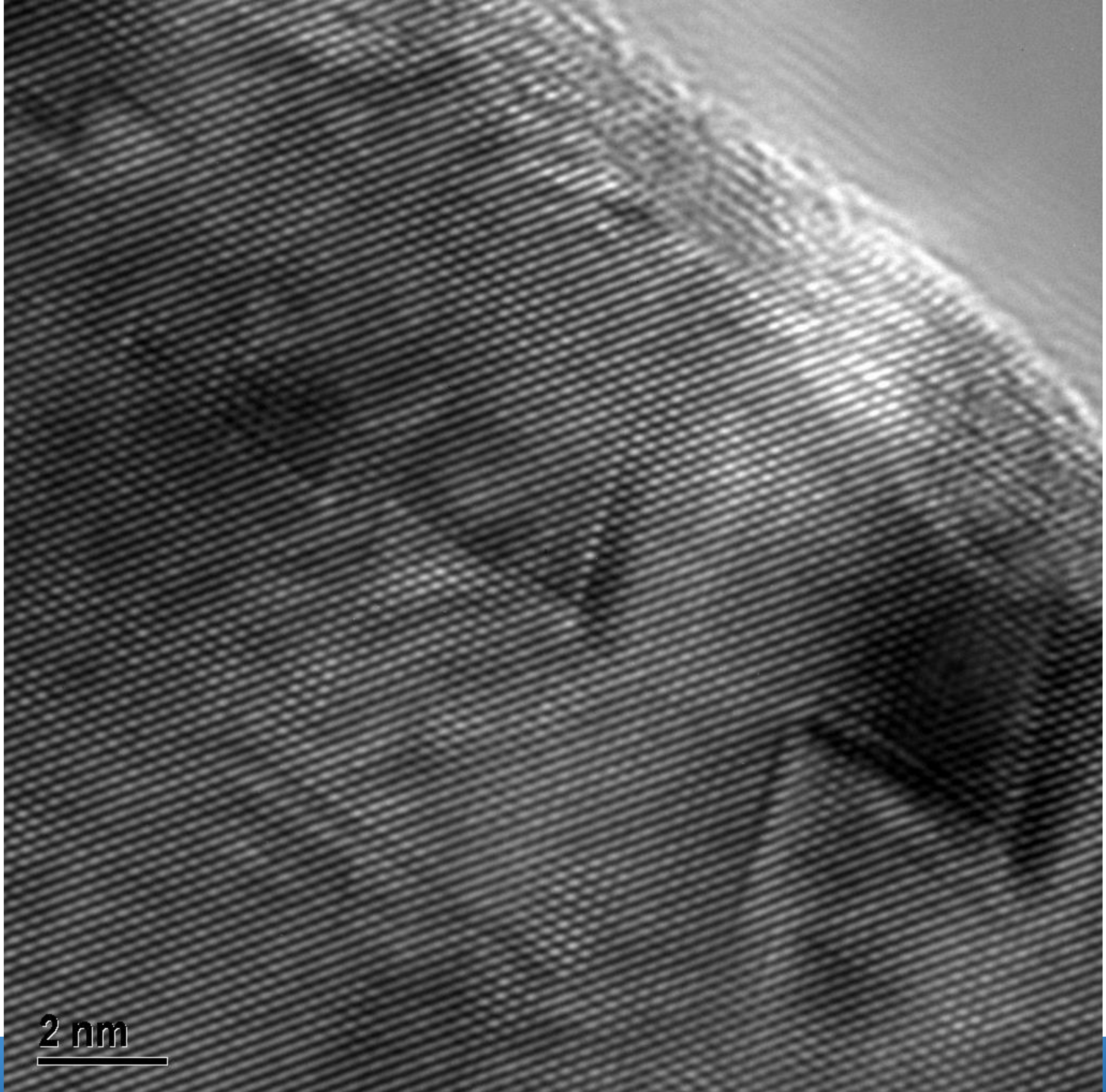
Need to go to higher values – but limited currently by conditioning! BD at 30-40 MV/m

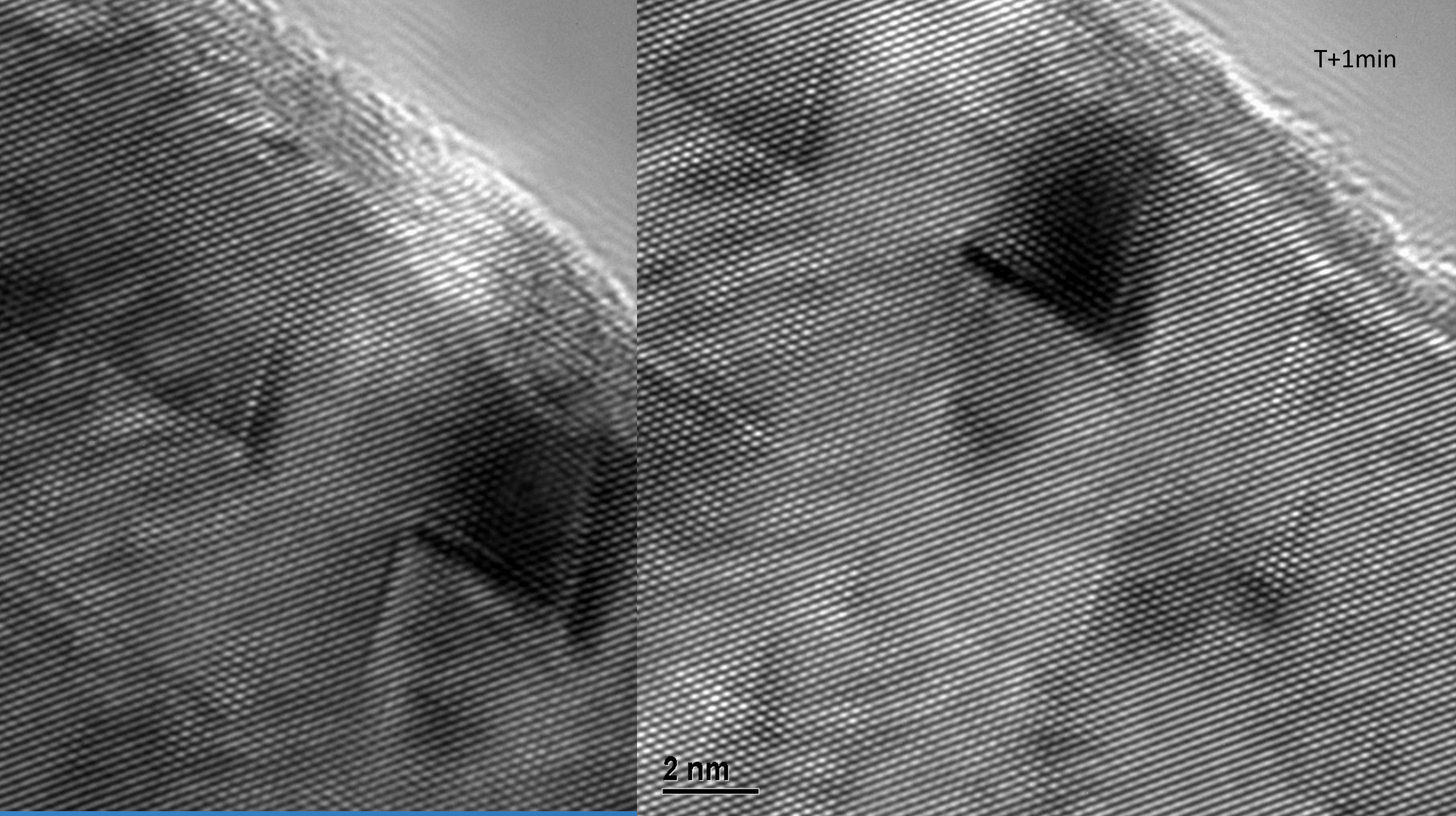
- Model - dislocations lead to critical transition
- Expected pre BD increase in fluctuations
- But still no dislocations specific signal.
- SO - Back to the microscope



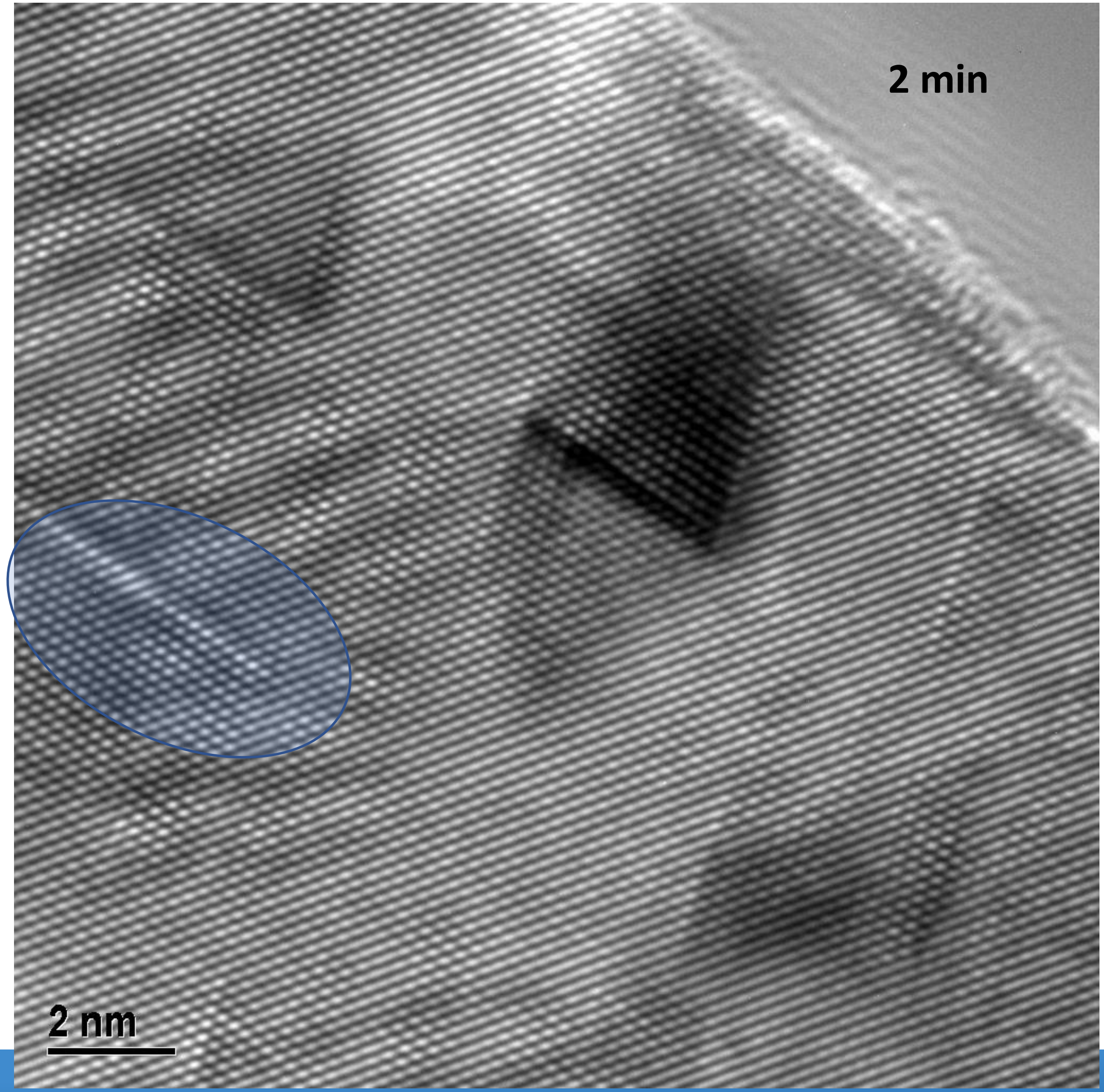
b=1/2[1-10]
b=1/6[-12-1]
Shockley partials
b=1/6[2-1-1]
Frank partials
b=1/3[1-11]
b=1/3[-111]
2-20
g
0.2 µm

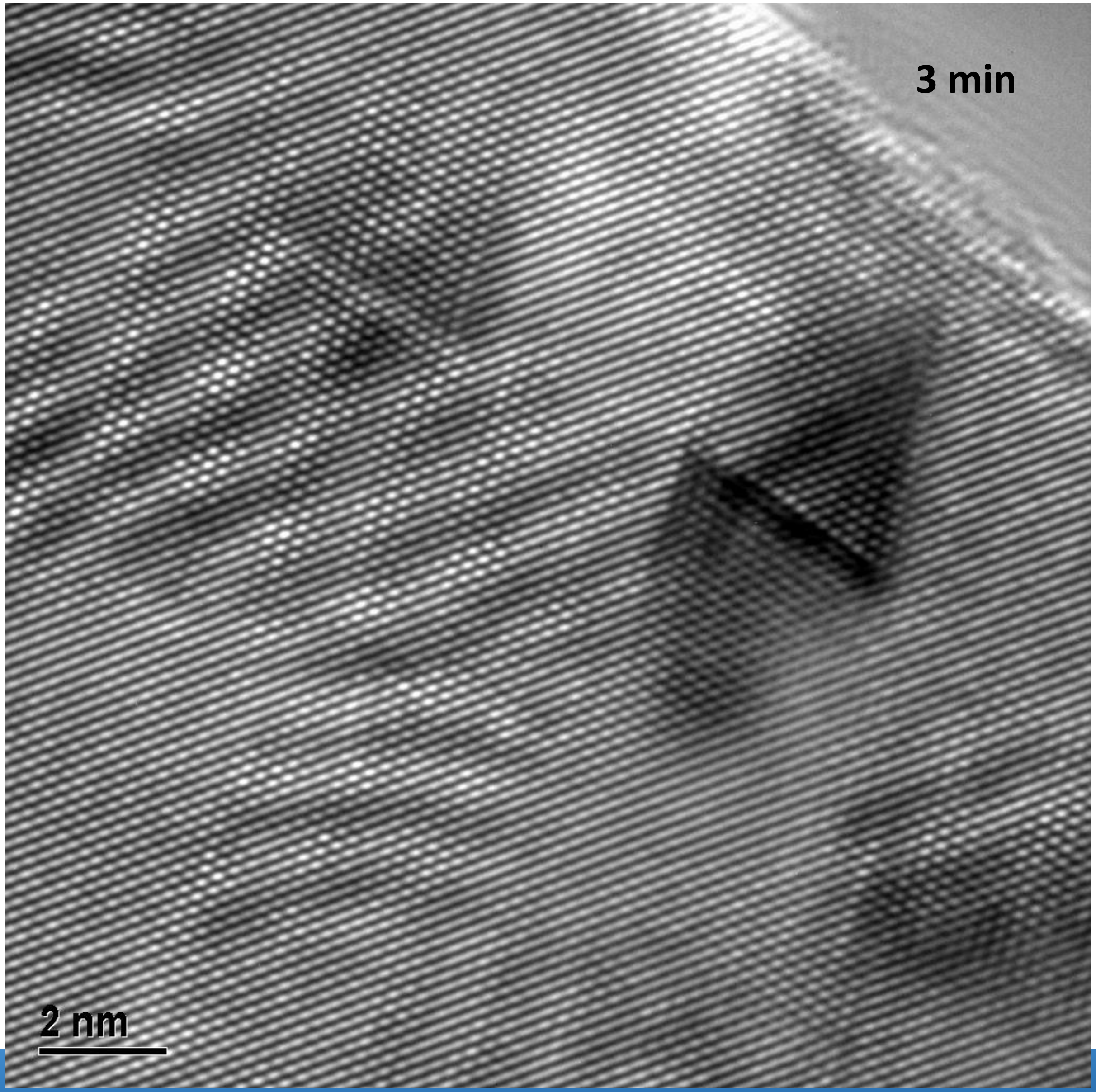

50 nm


2 nm

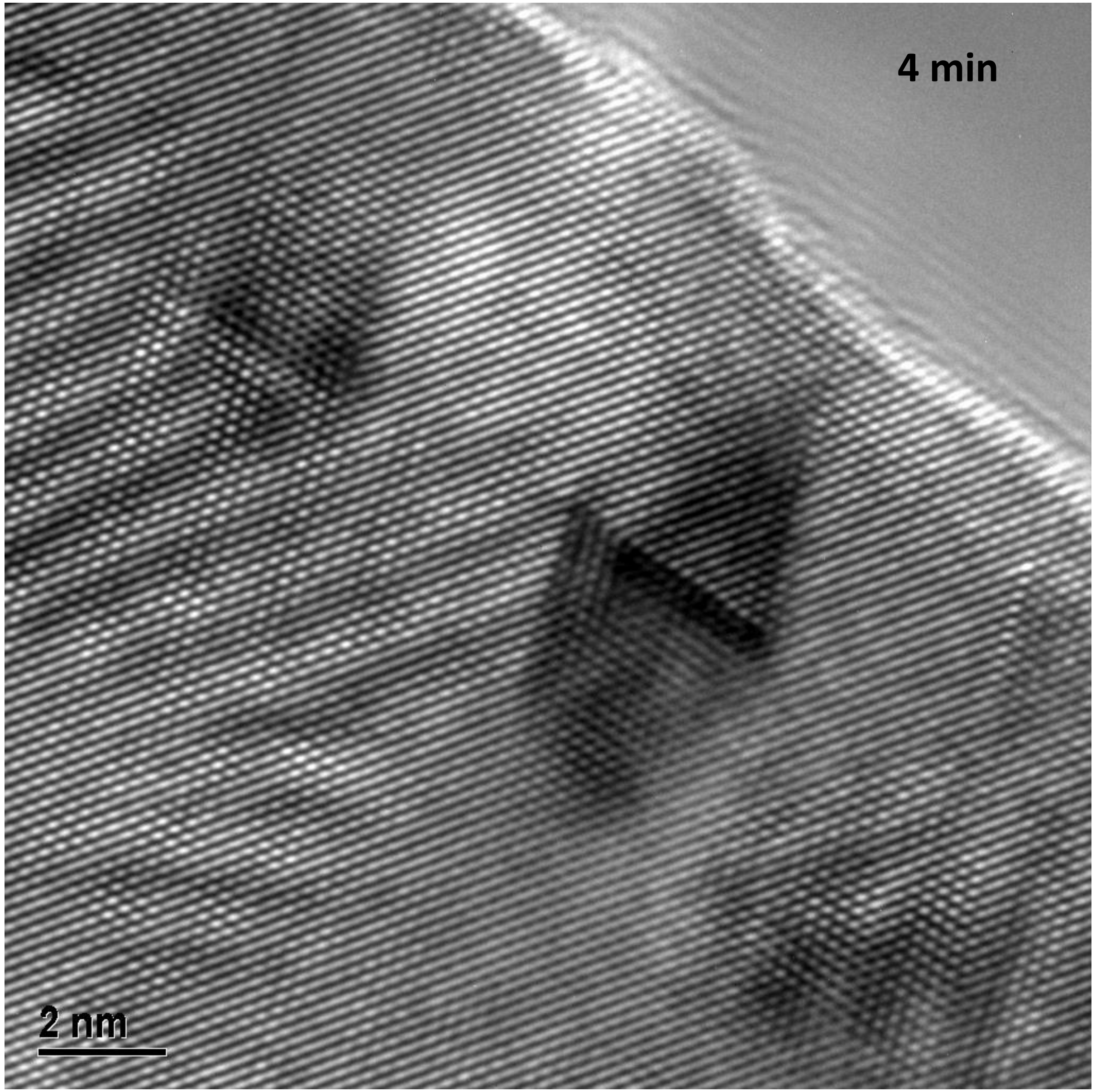

T+1min
2 nm

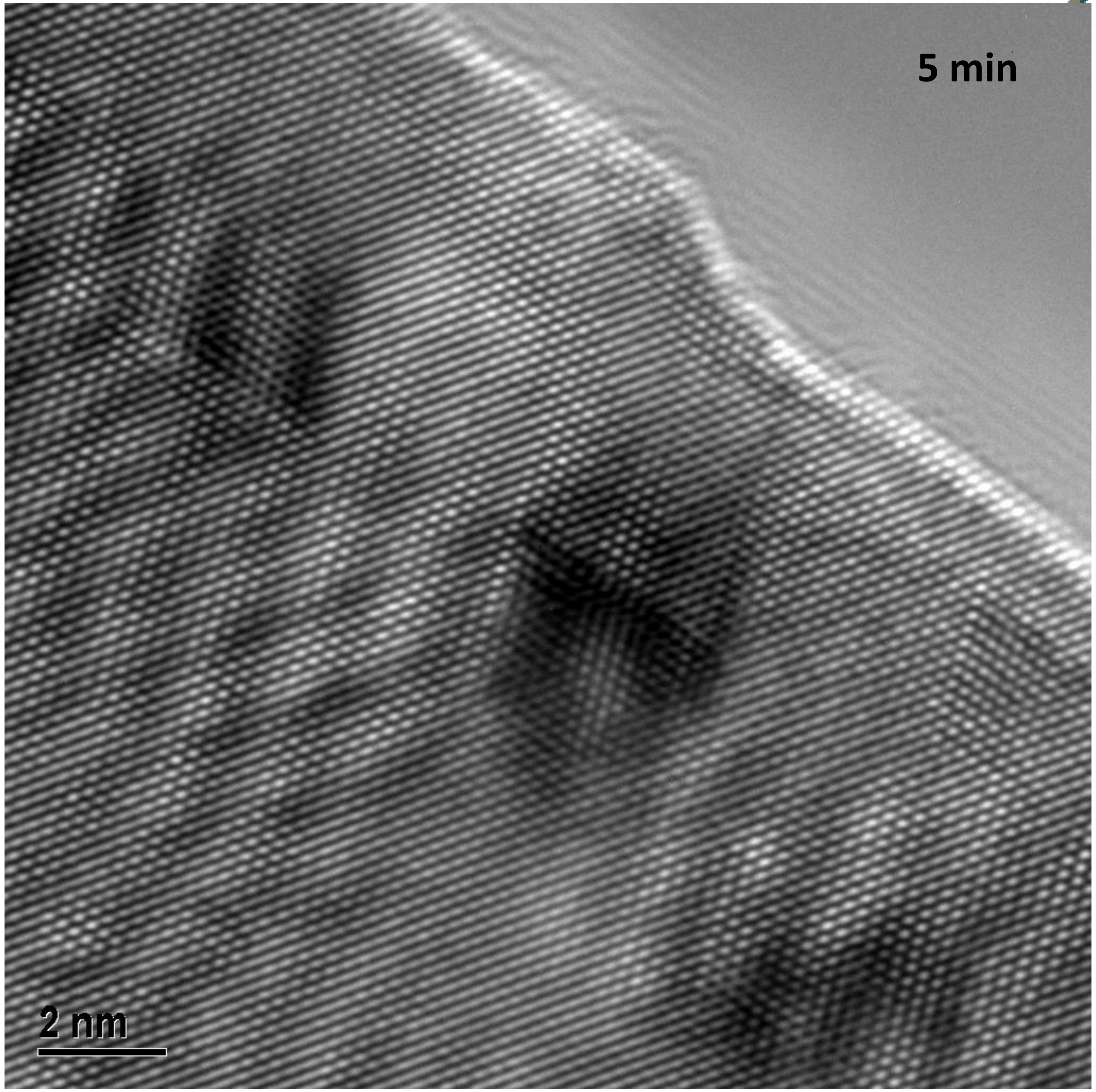
In situ observation : defects relax at free surface





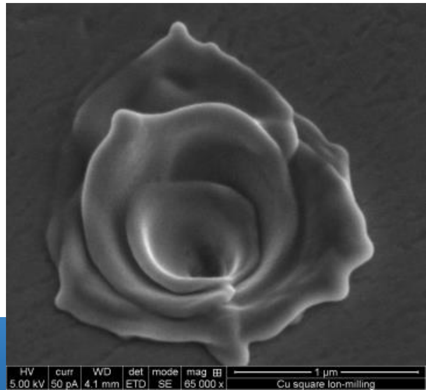
In situ observation : defects relax at free surface





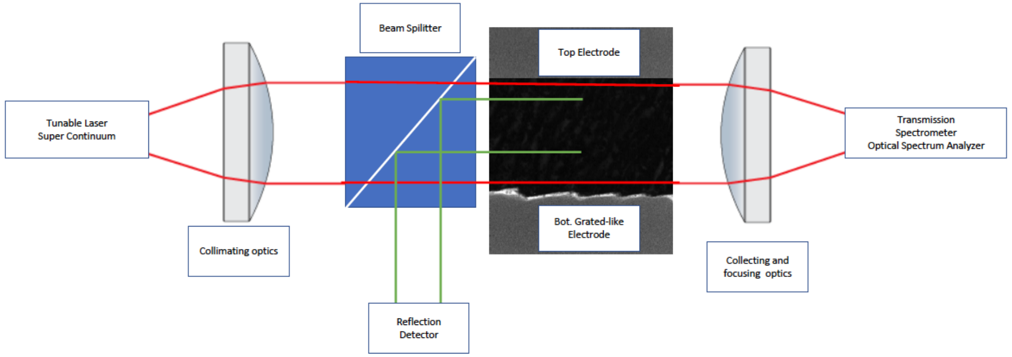
Summary and Outlook

- Electrodes maintain a highly ordered dislocations array.
- Proposed a direct link between plastic mechanism and BD nucleation:
  - Critical transition in the mobile dislocation population nucleates BD.
  - Dislocations show to move and modify surface even without external fields (at specific conditions)
  - Critical type fluctuations are observed for the first time in dark currents (Jan!)
- BUT:
  - Surface hardening seems to be not related to conditioning mechanism.
  - No clear modification of dislocation structure due to conditioning.
  - We still fail at measuring explicitly fluctuations in dislocations population
- Where can this take us:
  - Maybe effects are due to surface – electronic states interactions?
  - Plasmons?
  - Can we identify plasmons / surface evolution / acoustic emission?
- All this leads to
  - a proposal for a new experimental system – monitoring surfaces exposed to high fields via optics. (R. Gad and W. Wuensch)
  - Seeking for deeper understanding of observed structures
  - Continuing to study observed current fluctuations.
- Can you help? Yes!
  - Dedicated high sensitivity, high frequency, field emissions prior to BD Samples demonstrating strong variations in BD characteristics / conditioning



Basic concept for optical diagnostics for plasmonic-work function analysis

- Aiming at a new optical setup



- Manufactured acoustic resonator on the surface (grating)
- Aim at measuring: absorption spectra (plasmons?) as well refelction indicating surface evolution under external field.