Correlating Defect Microstructure and Hydrogen Retention in Supersaturated Layer of Tungsten Surface

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- Introduction: Super-Saturated Layer (SSL)
- Defect microstructure of SSL
- H depth profiling of SSL
- Conclusion

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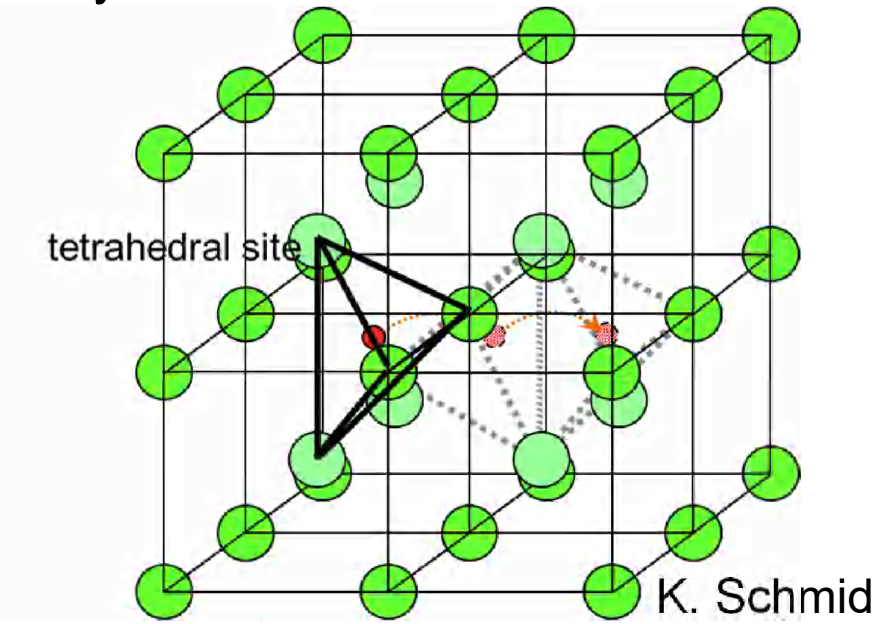
Introduction: H in W

- Hydrogen forms an interstitial solution in bcc W at **tetrahedral sites**
- Solute hydrogen isotopes (HI) atom **diffuses very fast** in W:
  D_{eff} ~ 10^{-7} m^2/s with low diffusion barrier (0.28 ± 0.06 eV[1])
- Crystal defects dominate HI retention in W: **rather low HI solubility**



W
- **Endothermic HI solution**
- **No hydride forming**

HI loading

Trapping by either intrinsic or Irradiaiton-induced defects

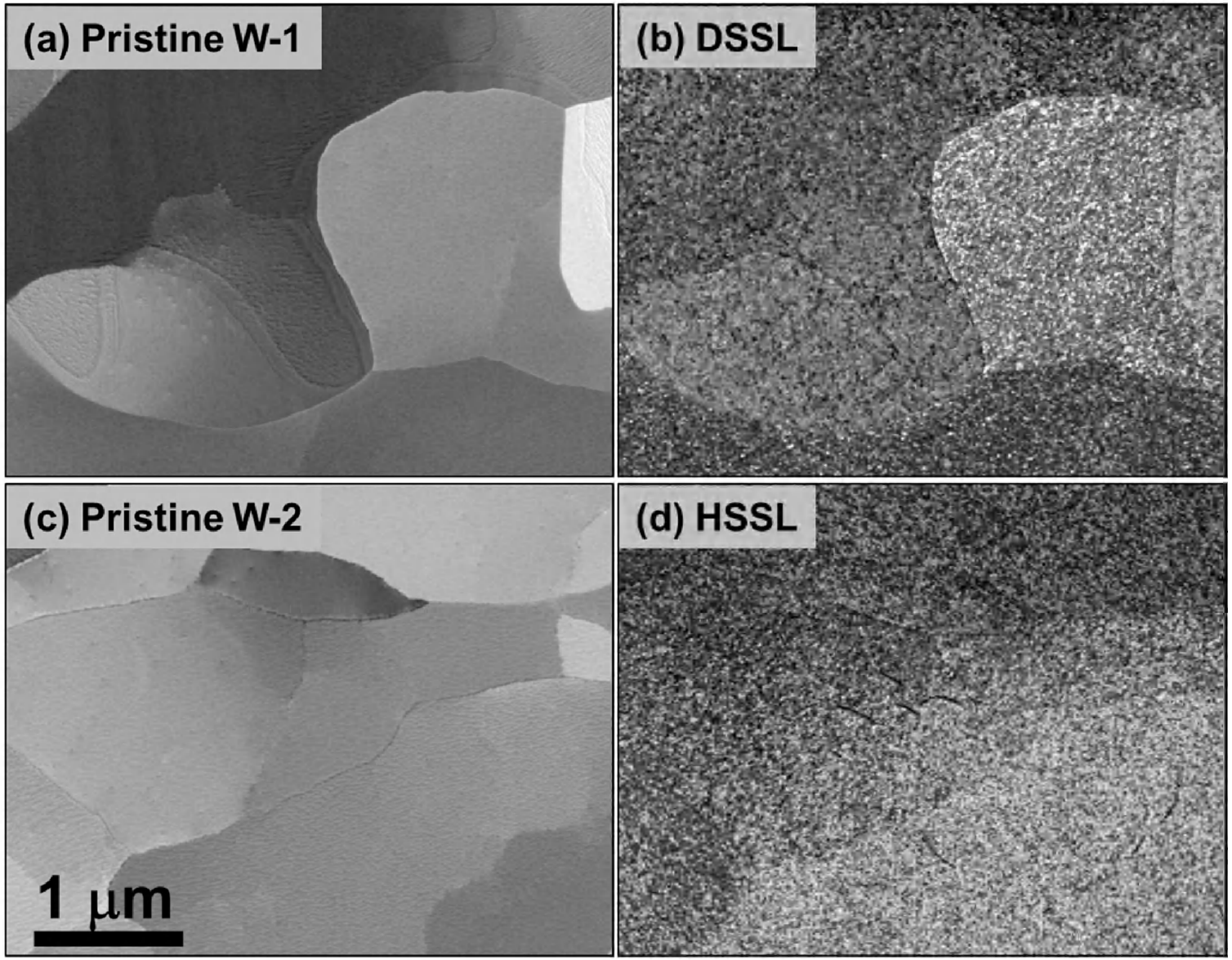
Kinetic W damage threshold (stable Frenkel pair formation): 40-70 eV[2,3]

W of 40 eV ←→ D with ~ 920 eV

H with ~ 1840 eV

[1] G. Holzner *et al.*, *Phys Scripta, **T171** (2020) 014034*
*[2] F Maury et al., 1978, Radiation Effects **38**, 53*
[3] K Nordlund et al., 2015, Primary Radiation Damage in Materials.

Sub-threshold damage:SSL



SSL: Super-Saturated Layer

Plasma exposure: 300 K, 6e24/m²

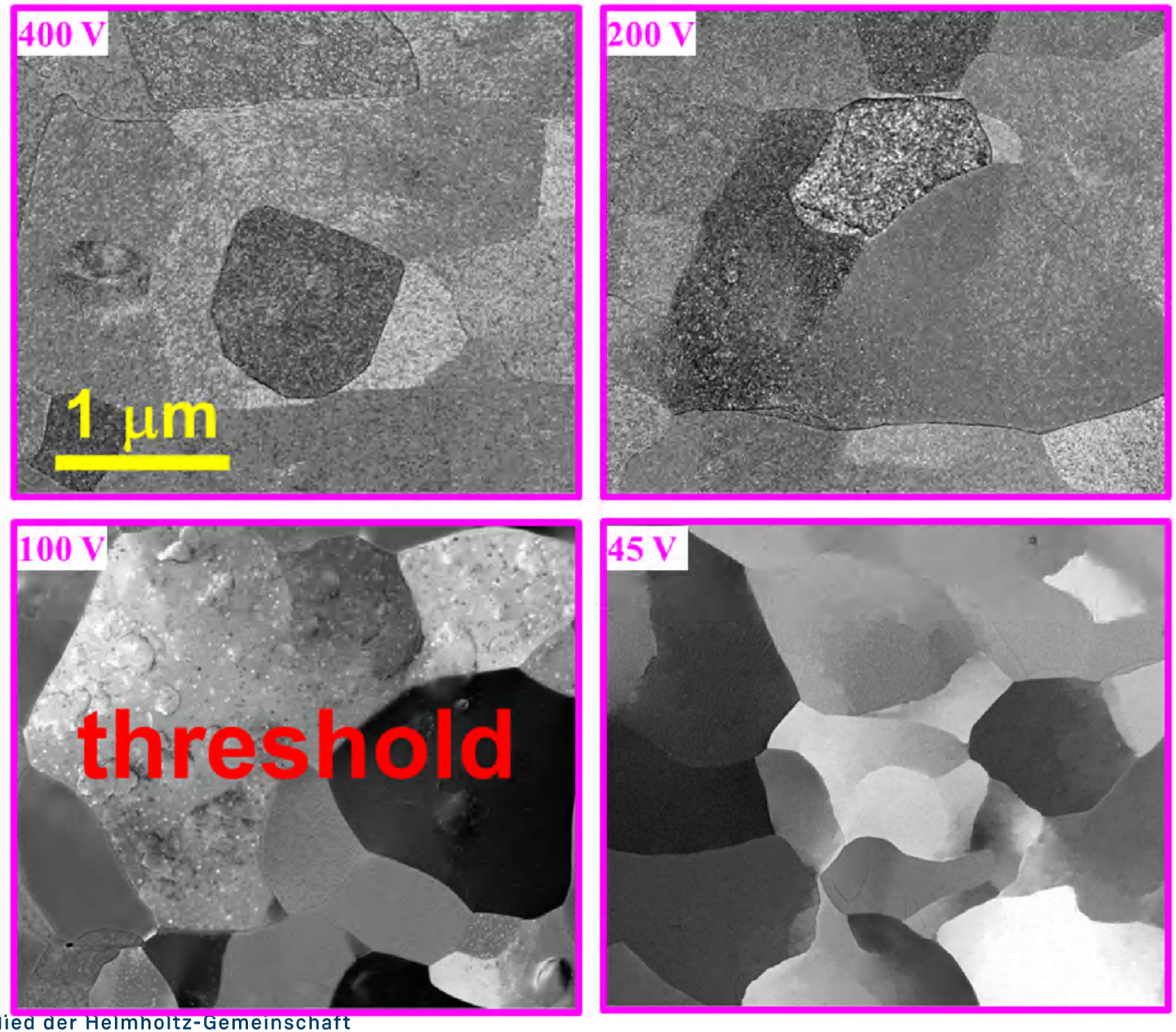
D: 215 eV / H: 415 eV

<< E_{th} for stable FP formation (FP = Frenkel Pair)

Strong surface modification due to SSL with extended defect structures rather than single vacancies from the primarily created FPs

*[1] L. Gao et al., Nucl. Fusion **57** (2017), 016026*
*[2] L. Gao et al., Acta Mater. **201** (2020) 55–62*

SSL: Ion energy thresthold



300 K, 1×10^{24} D/m^2

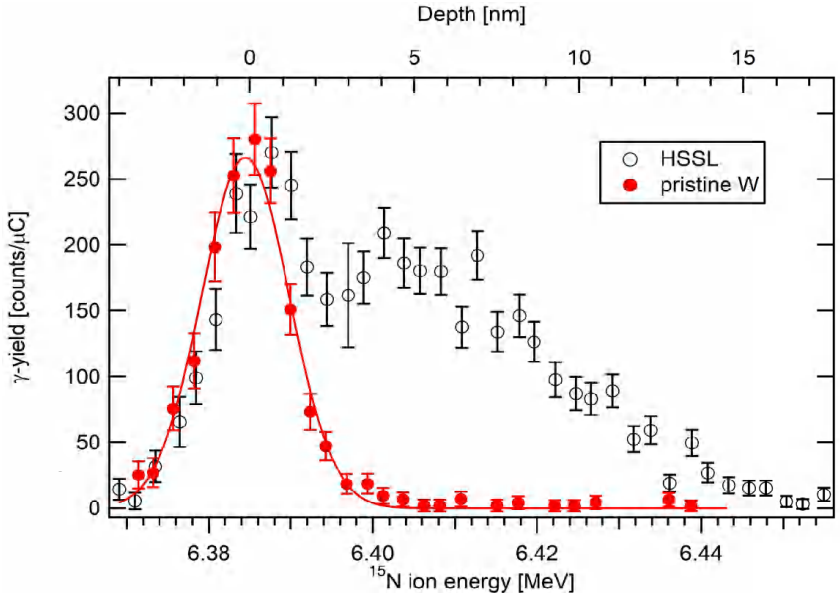
- At bias above 100 V, all grains distorted
- At 100 V bias, only some grains distorted
- At bias below 100 V, no visible damage

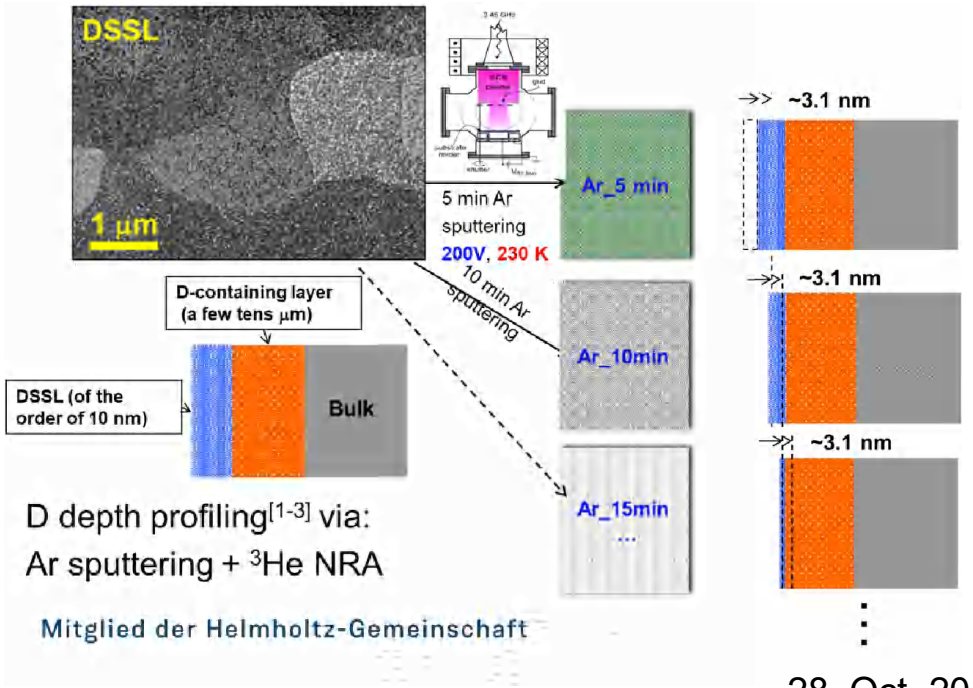
➢ **115 eV**: energy threshold for damage production in SSL

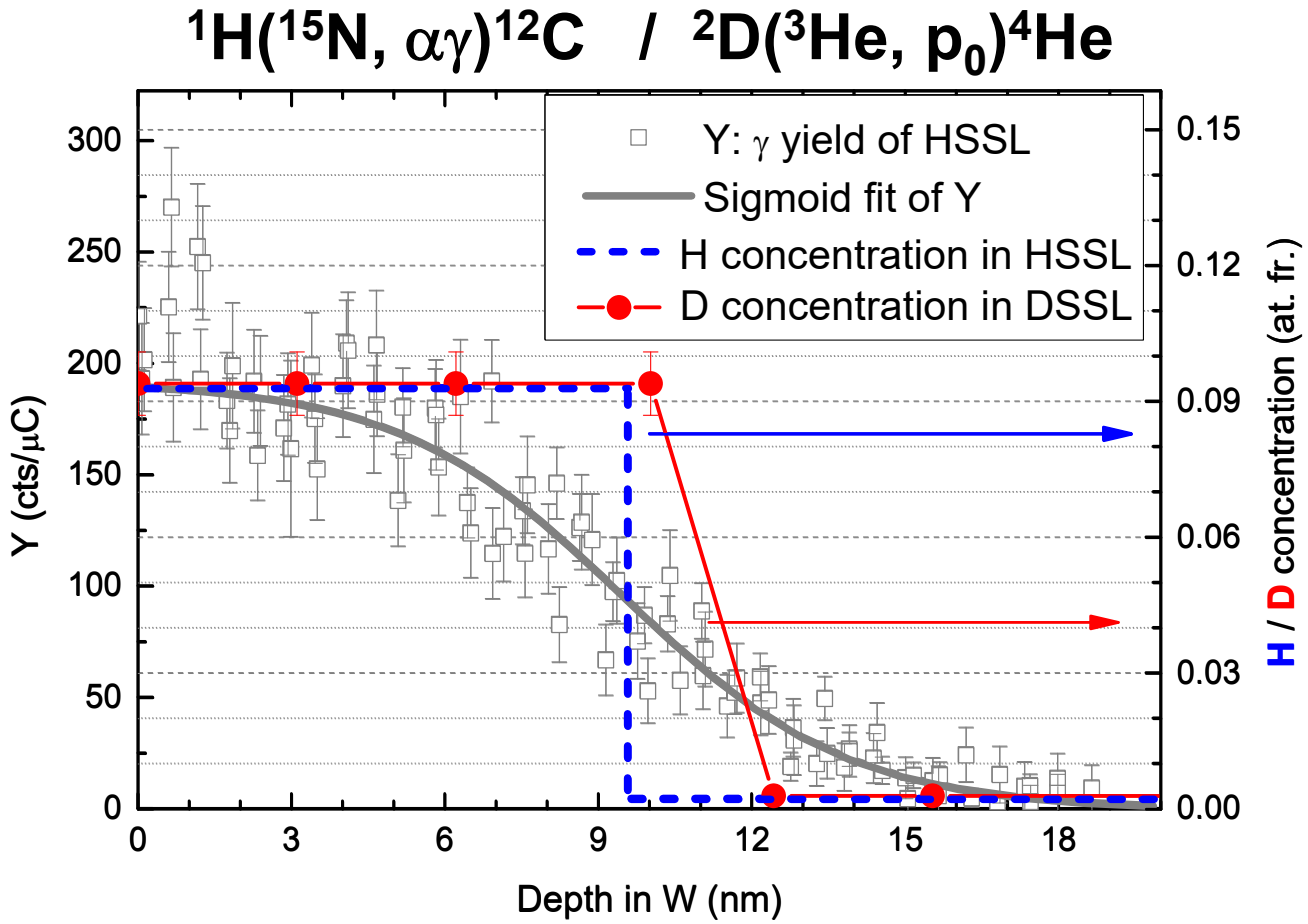
→ **5eV** transferred to W atom

*[1] Gao et al., Nucl. Fusion **57** (2017), 016026*

H/D Supersaturation measured







*[1] L. Gao et al., Nucl. Fusion **57** (2017), 016026.*
*[2] L. Gao et al., Acta Mater. **201** (2020) 55–62*

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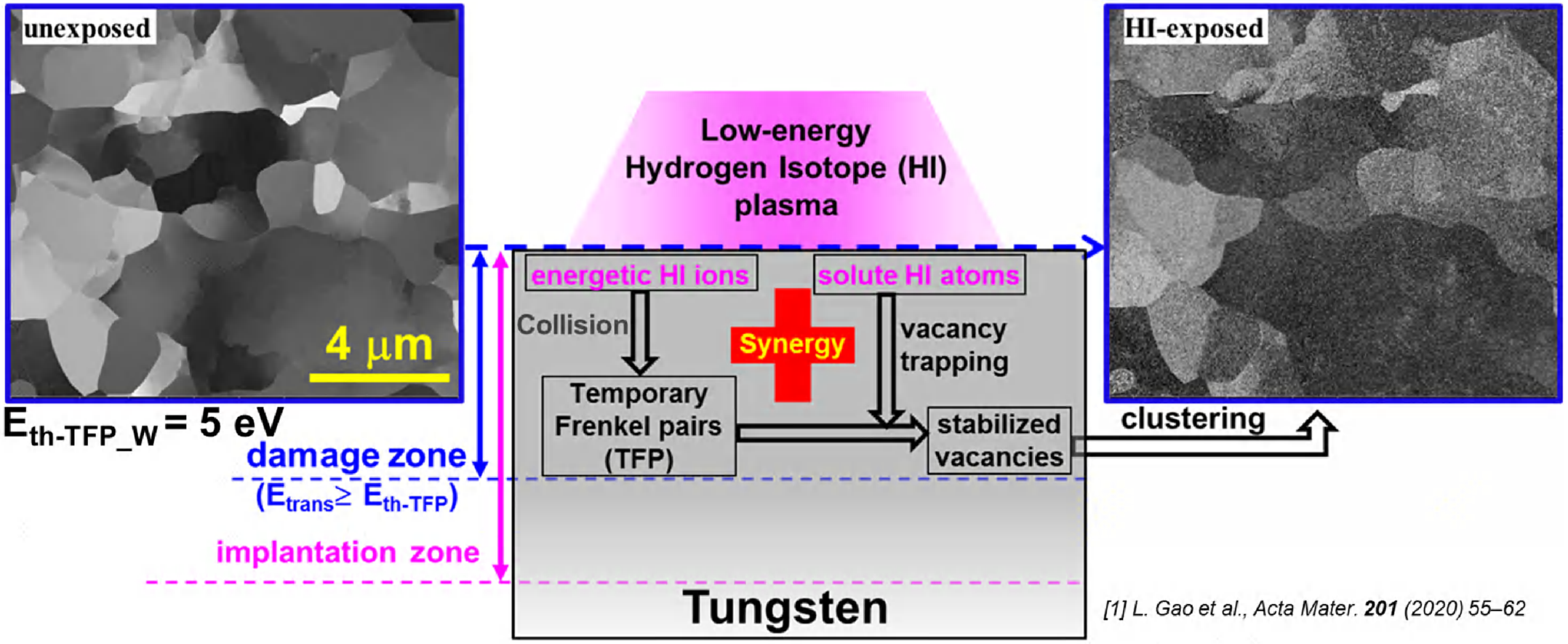
SSL: physical model

SSL formation by HI atom-ion synergy at sub-threshold ion energy[1]

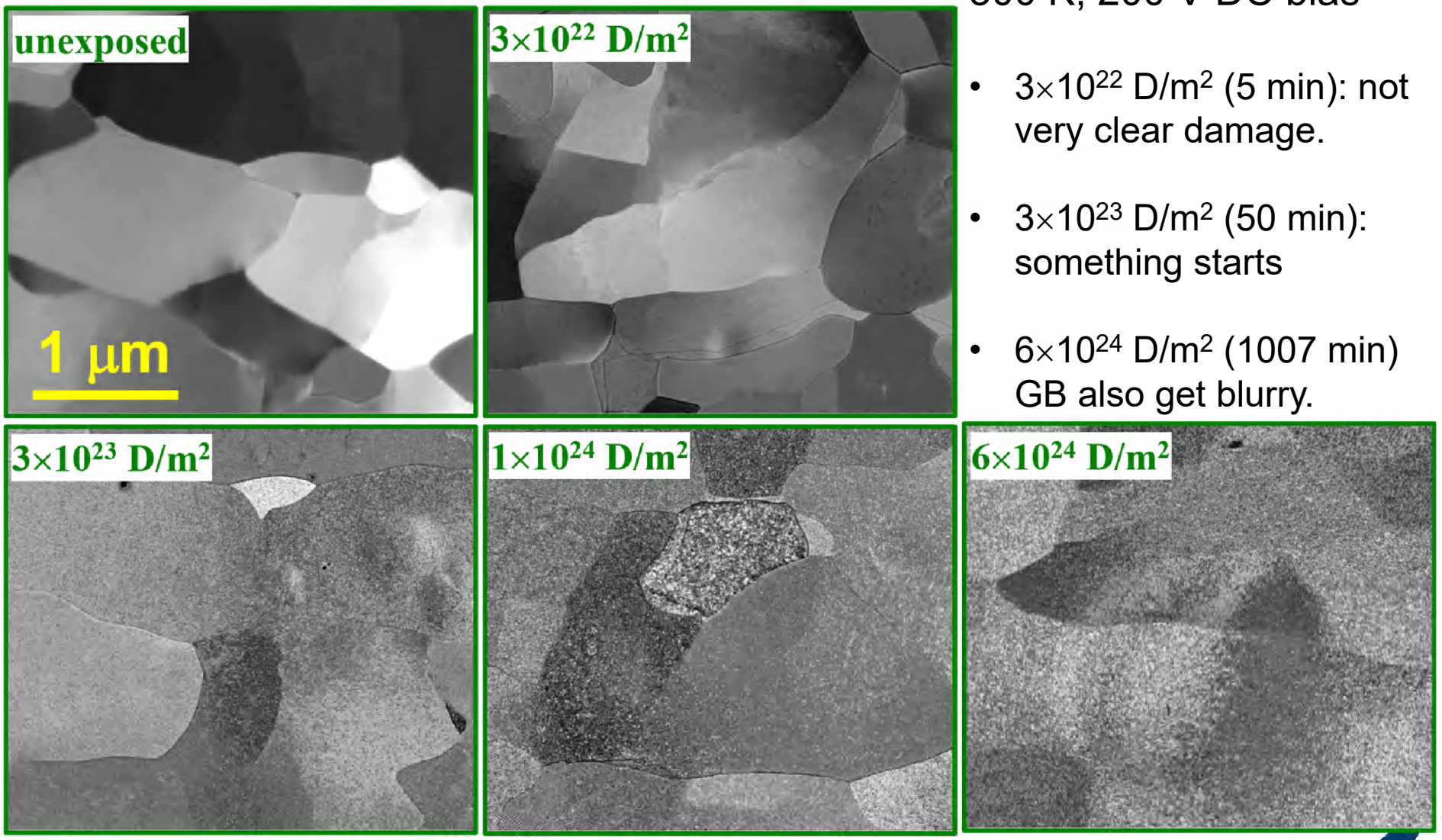


[1] L. Gao et al., Acta Mater. **201** (2020) 55–62

What defect microstructure can hold ~10 at.% HI concentration?

DSSL: fluence dependence

D fluence-dependent: development of DSSL



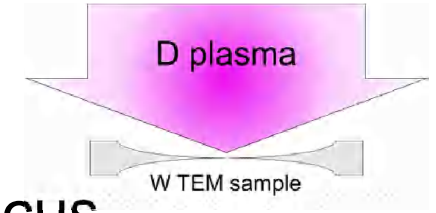
300 K, 200 V DC bias

- 3×10^{22} D/m^2 (5 min): not very clear damage.
- 3×10^{23} D/m^2 (50 min): something starts
- 6×10^{24} D/m^2 (1007 min) GB also get blurry.

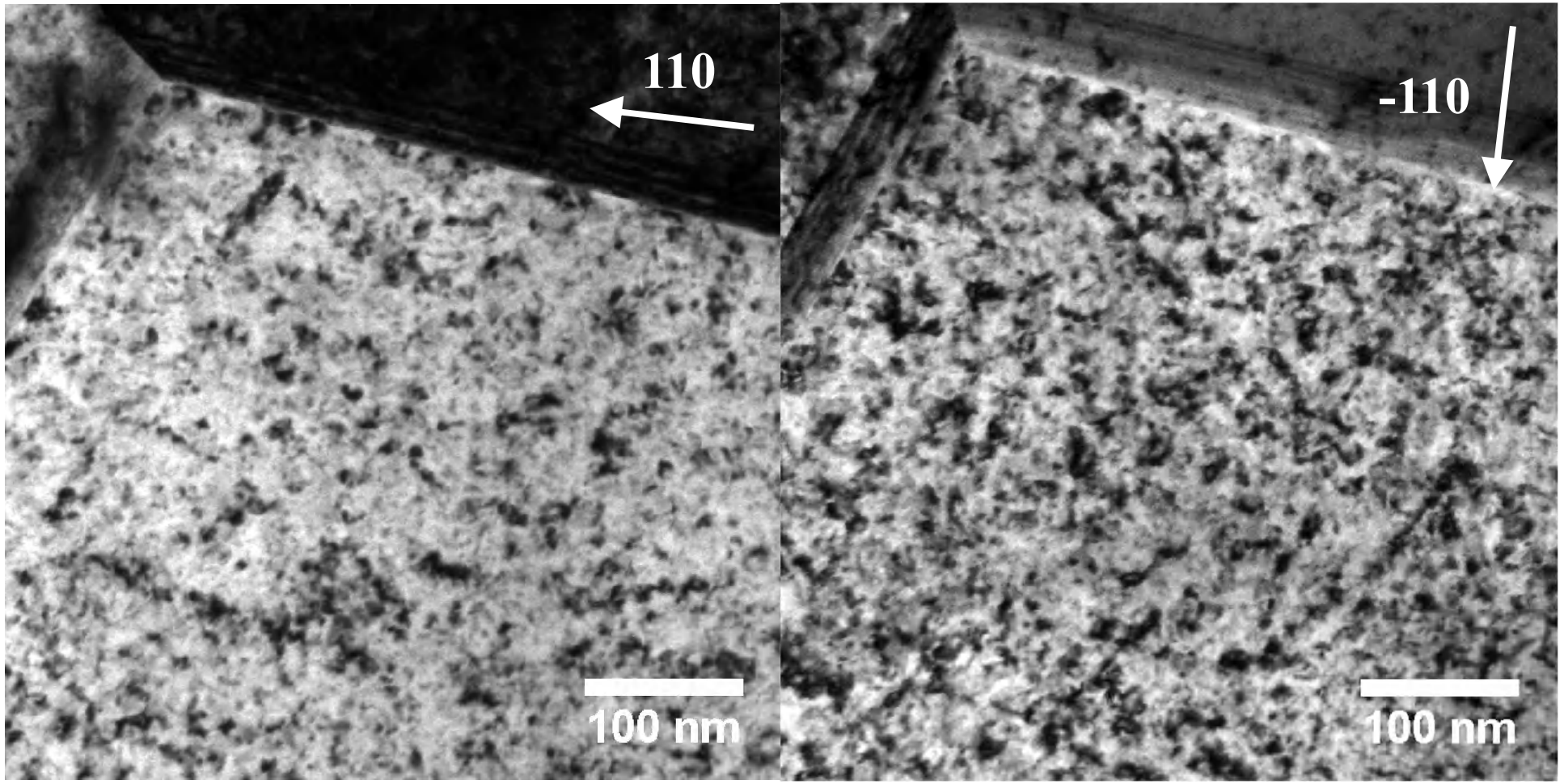
[1] L. Gao et al., Acta Mater. **201** (2020) 55–62

DSSL: defect microstructure by TEM

6×10^{24} D/m^2 (215 eV/ion, 300 K)



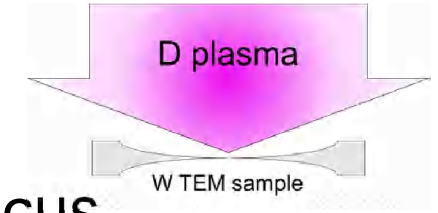
Thin region, in-focus



A high density of black spots (dislocation loops?)

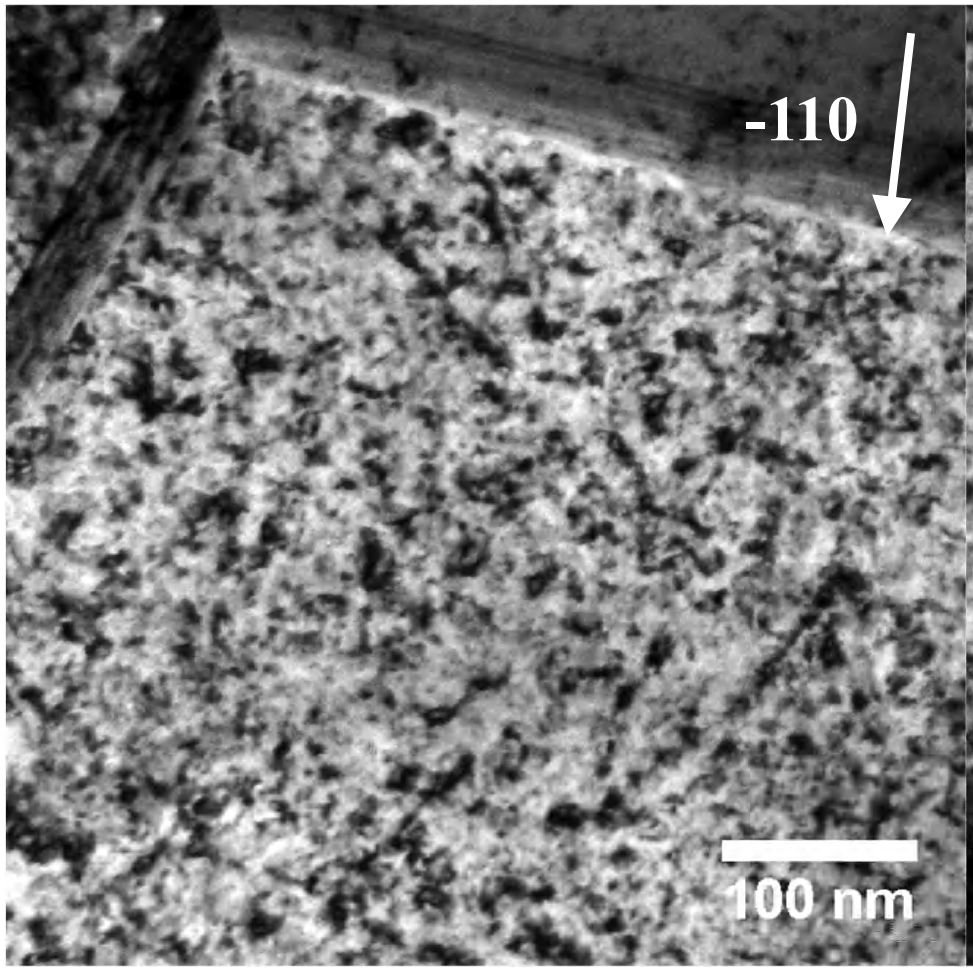
DSSL: defect microstructure by TEM

6×10^{24} D/m^2 (215 eV/ion, 300 K

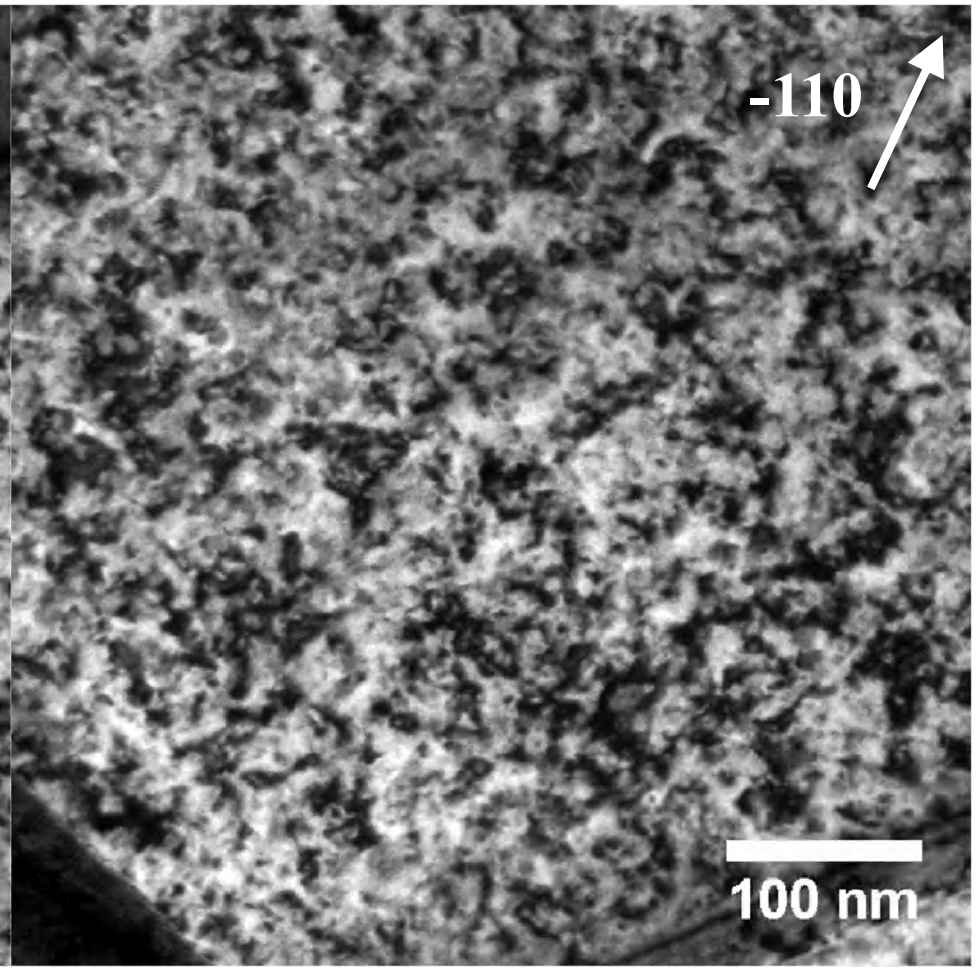


in-focus

ROI – thin



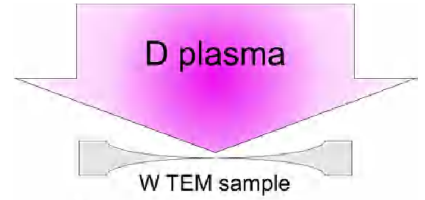
ROI – thick



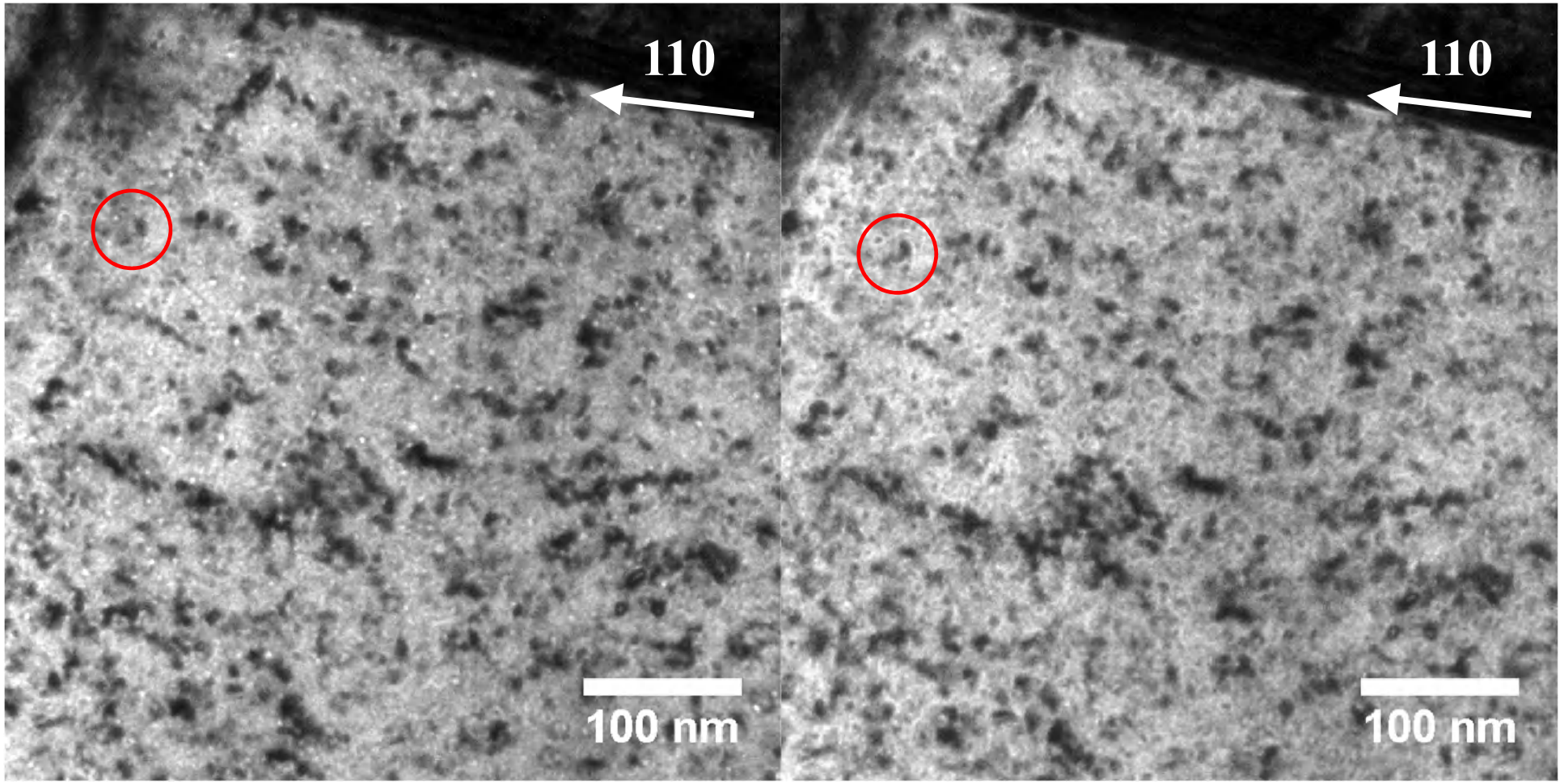
Damage structure appears thickness dependent

DSSL: defect microstructure by TEM

6×10^{24} D/m^2 (215 eV/ion, 300 K)



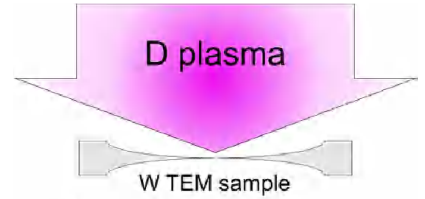
Thin region



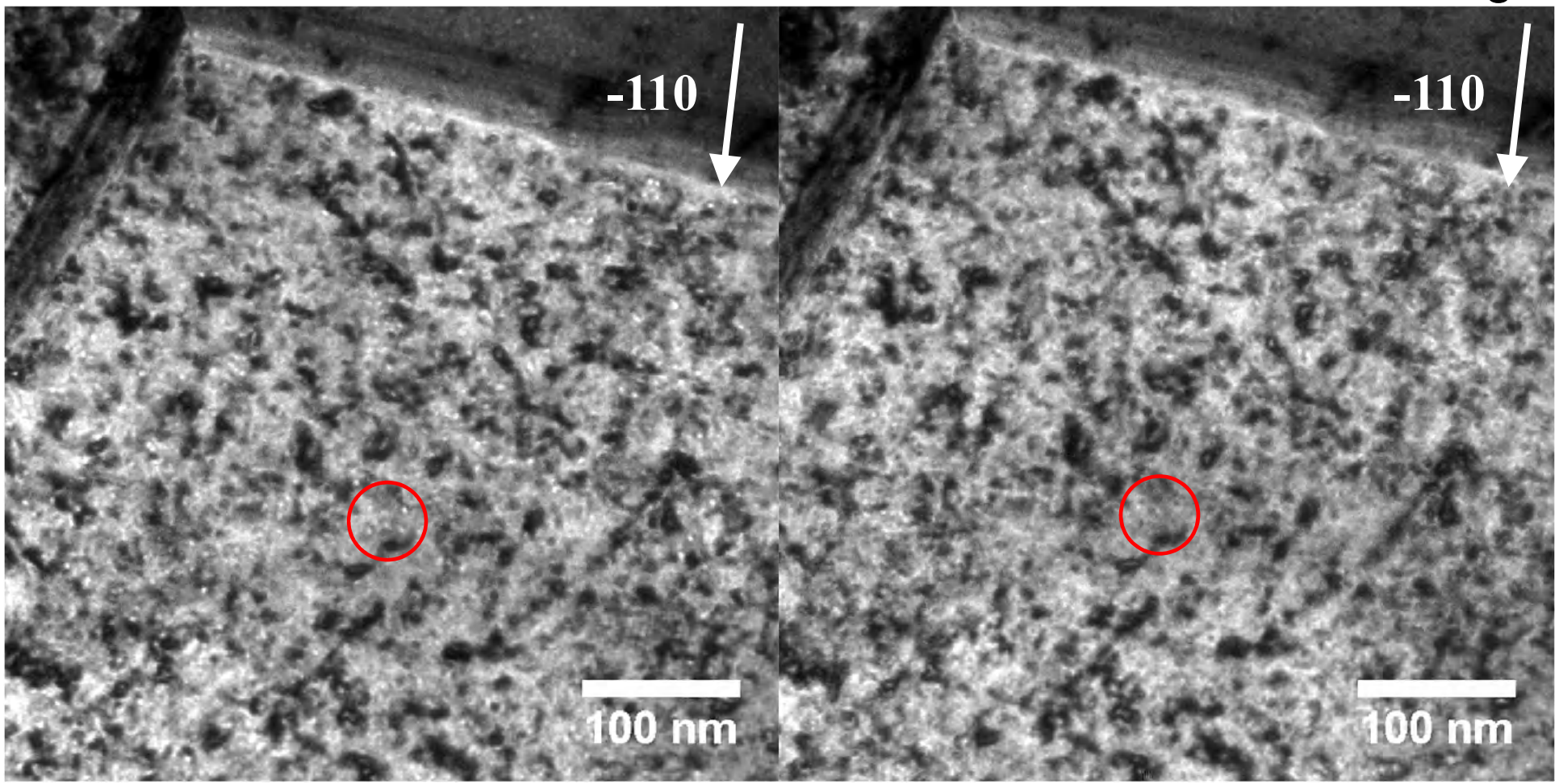
Presence of H-bubbles suspected!

DSSL: defect microstructure by TEM

6×10^{24} D/m^2 (215 eV/ion, 300 K



$\Delta f = -3.8\ \mu m$ $\Delta f = +3.8\ \mu m$ Thin region



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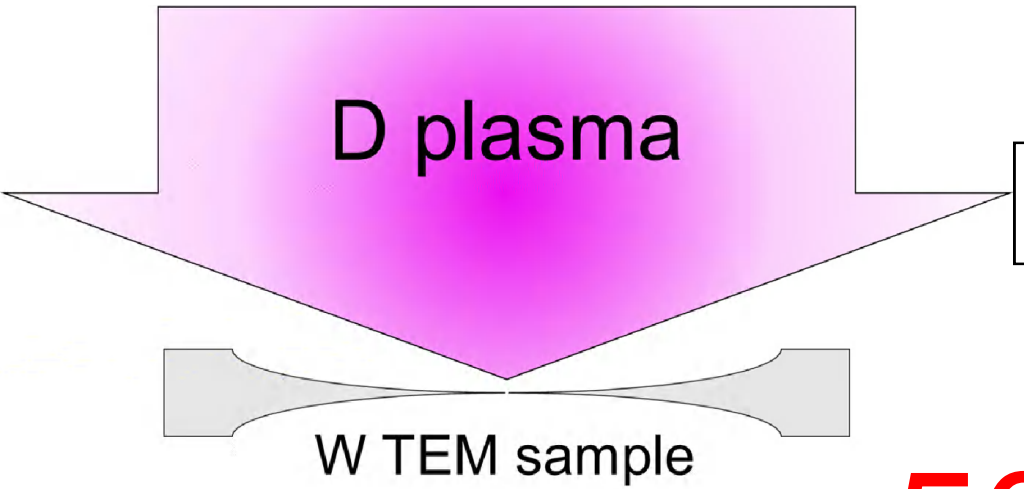
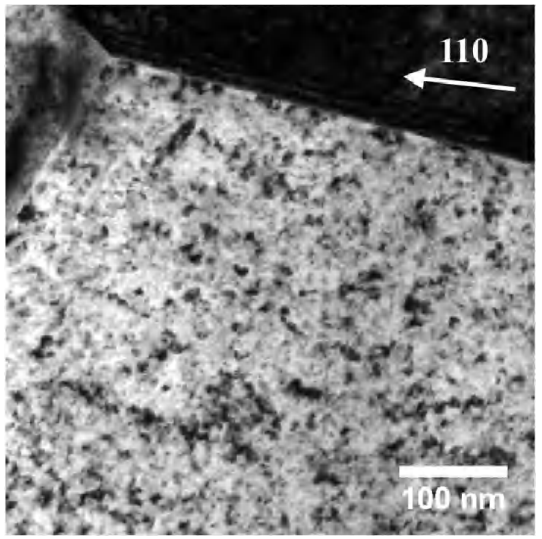
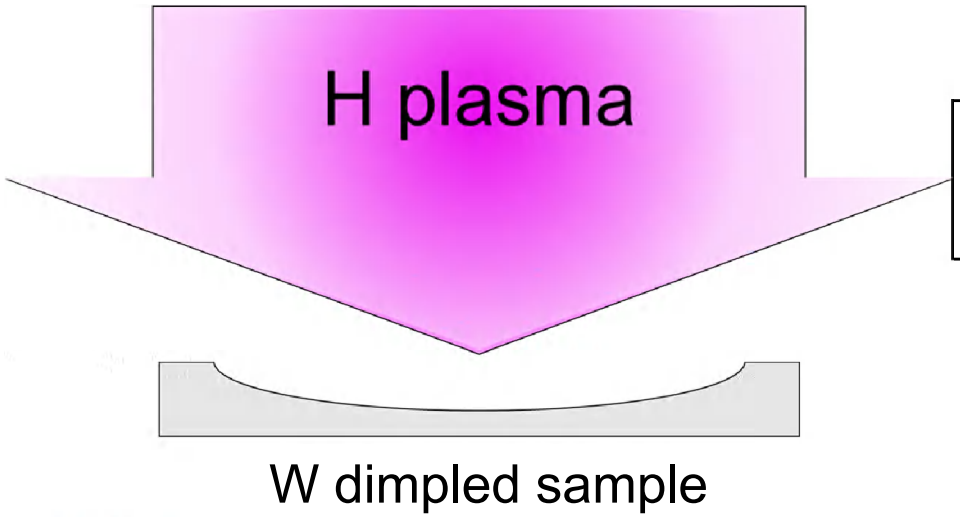
SSL: defect microstructure by TEM



TEM



50 Years!

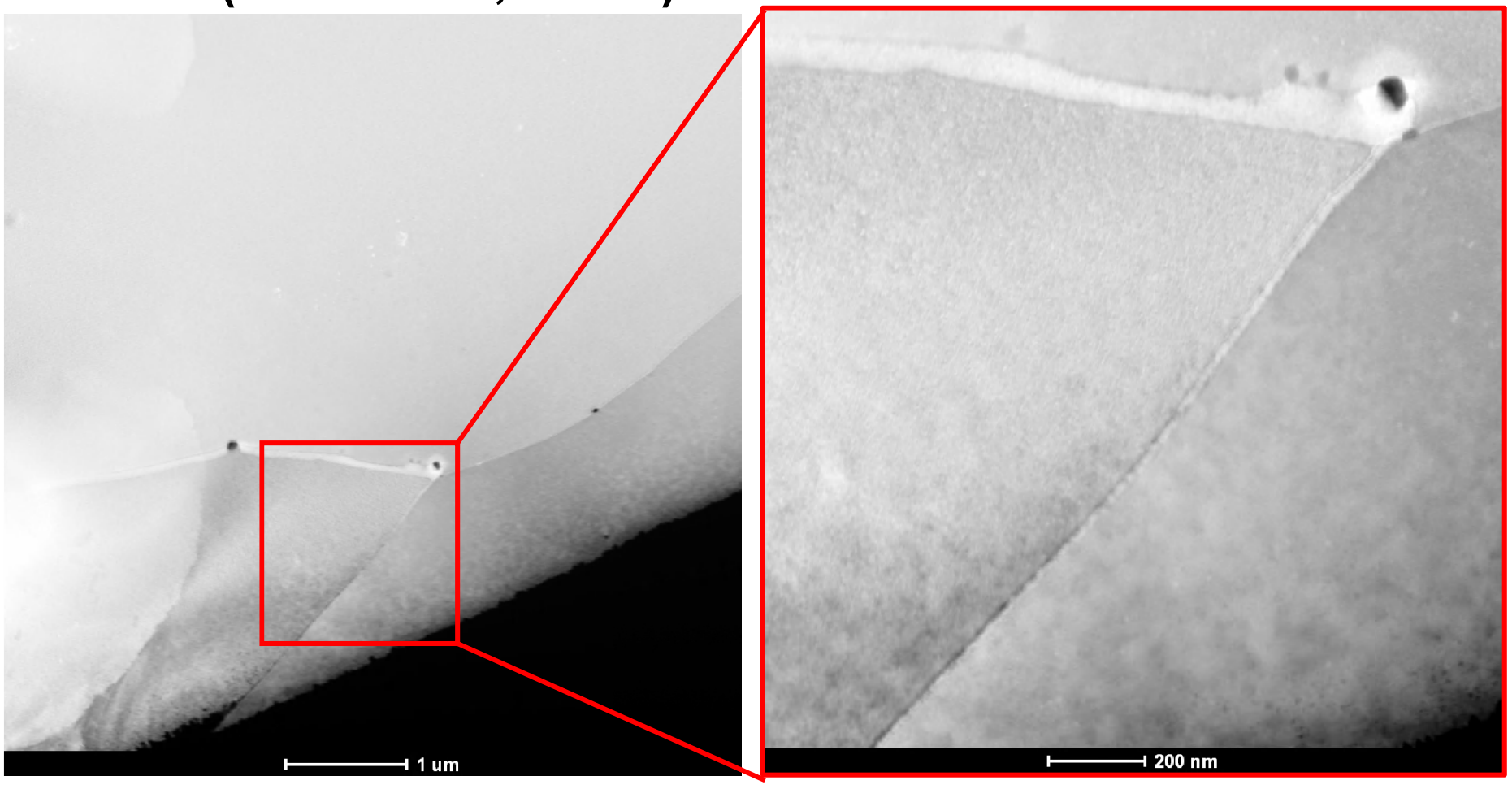


Electropolishing
(Back-thinning mode)

TEM
characterization

HSSL: defect microstructure by TEM

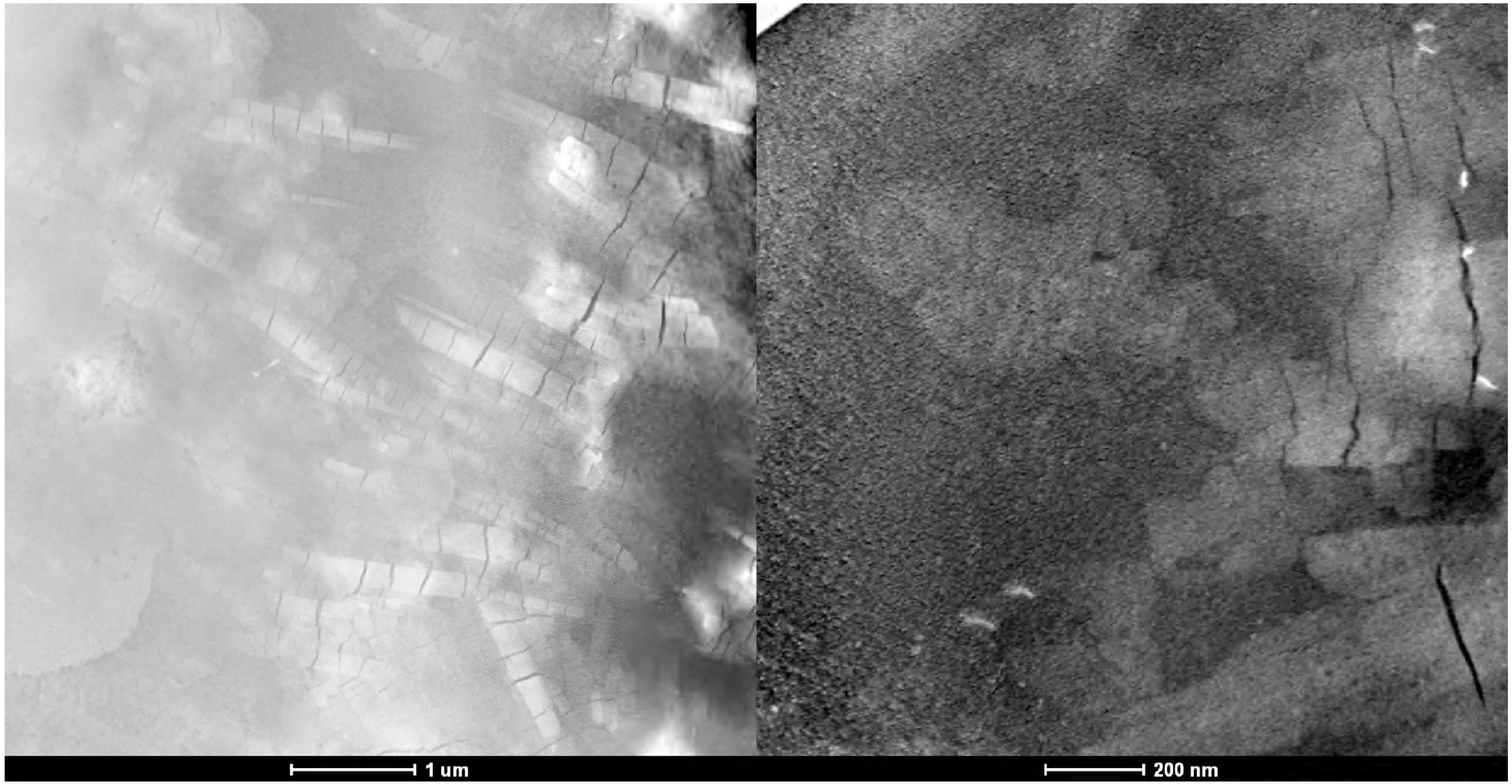
5×10^{22} H/m^2 (415 eV/ion, 300 K)



- Specimen overview (HAADF-STEM).

HSSL: defect microstructure by TEM

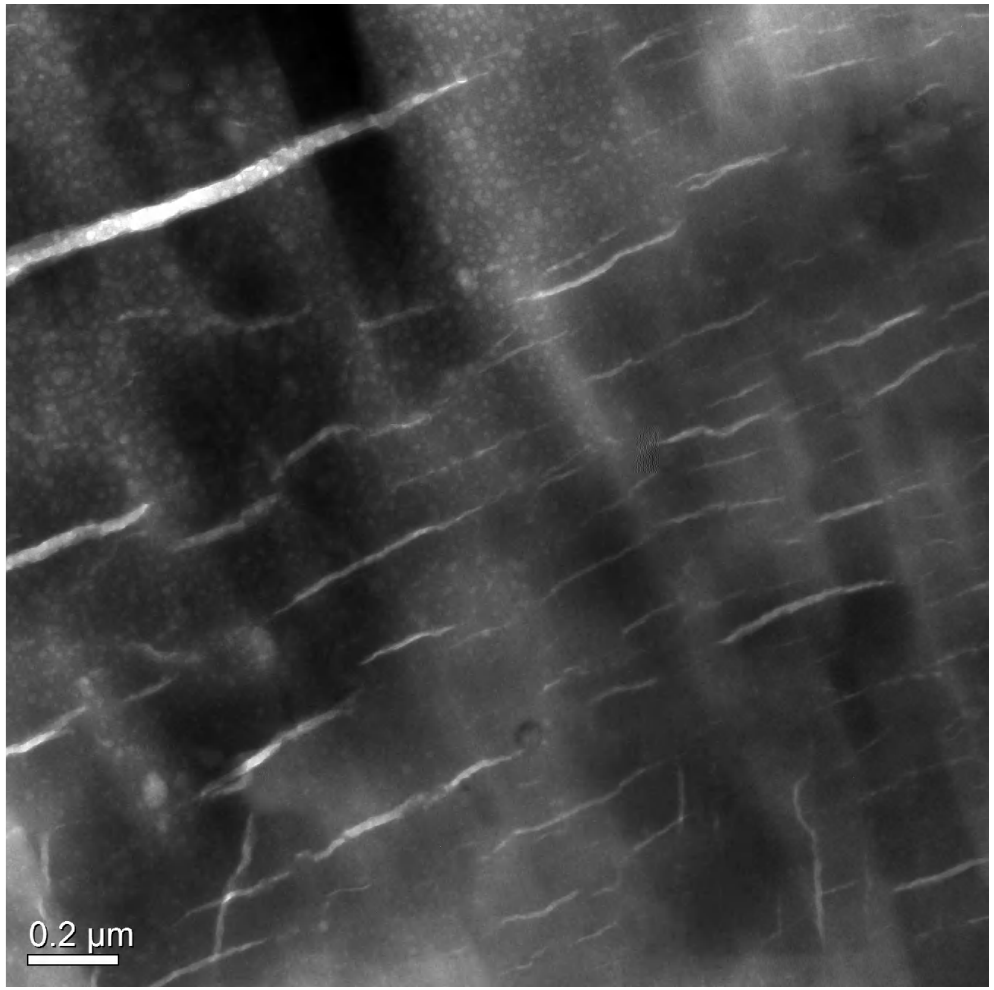
5×10^{22} H/m^2 (415 eV/ion, 300 K)



- Cracks develop within 1 week of thin-foil perforation treatment.
- HAADF-STEM.

HSSL: defect microstructure by TEM

5×10^{22} H/m^2 (415 eV/ion, 300 K)



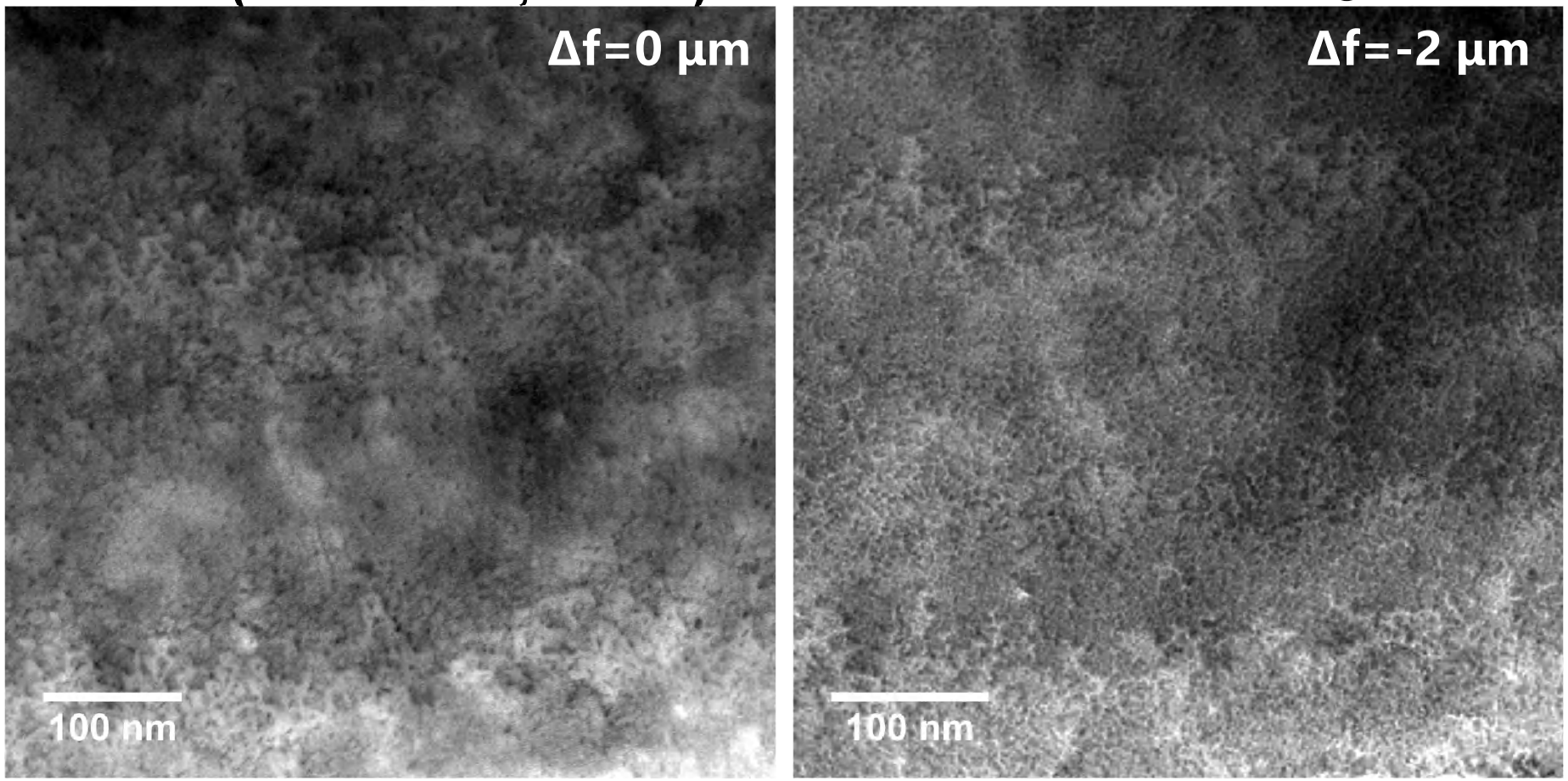
Thin region, in-focus

- Specimen overview (TEM-BF).
- Cracks develop within grains, initiation typically not associated with GBs.
- Densely populated H bubbles are embedded in these cracks.

HSSL: defect microstructure by TEM

5×10^{22} H/m^2 (415 eV/ion, 300 K)

Thin region

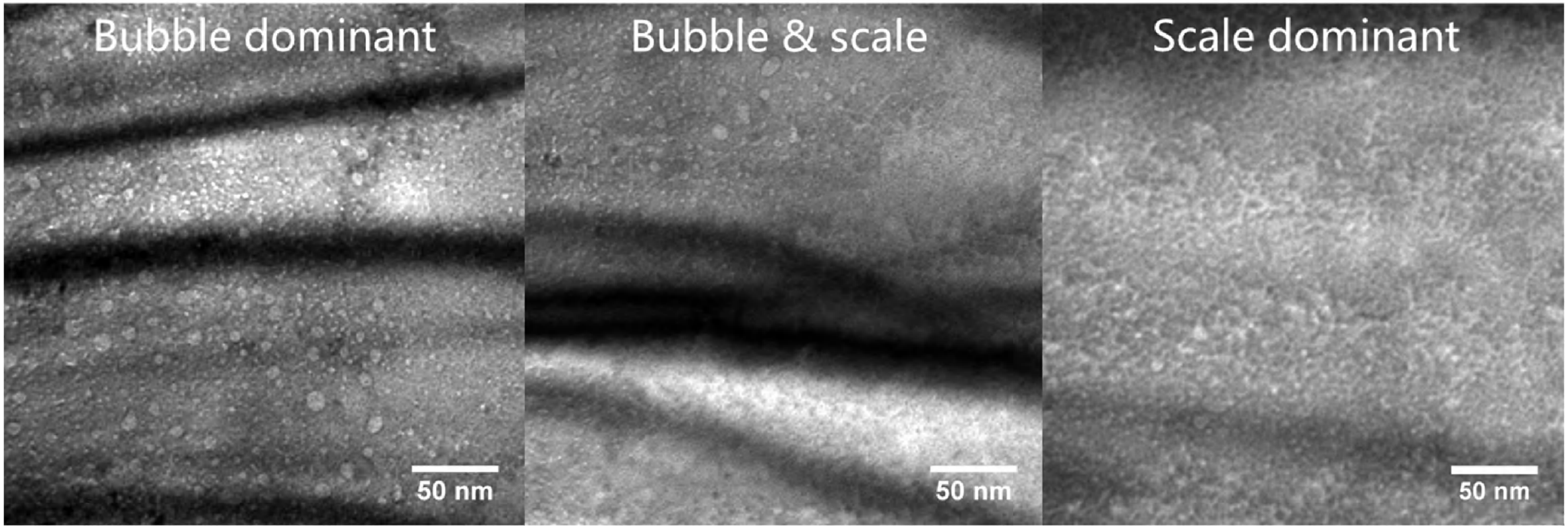


- Electron beam exposure (<3 min) (thickness<100 nm).
- "Surface scale" features dominate the microstructure: H-bubble rupture?
- Insufficient evidence upon presence of black-spot damage.

HSSL: defect microstructure by TEM

5×10^{22} H/m^2 (415 eV/ion, 300 K)

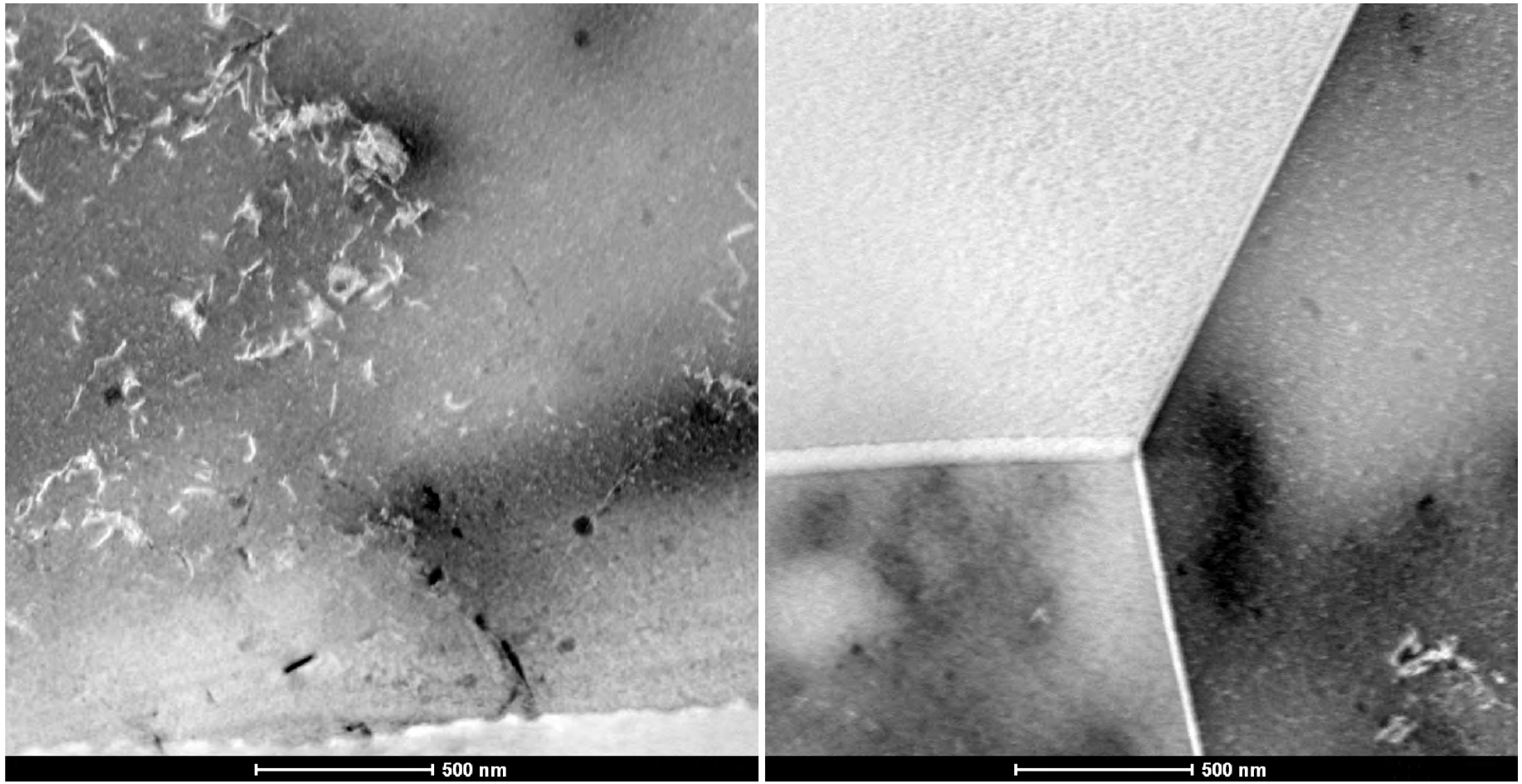
Thick region



- Electron beam exposure (<3 min) (thickness>100 nm).
- H bubbles rupture under e-beam irradiation

HSSL: defect microstructure by TEM

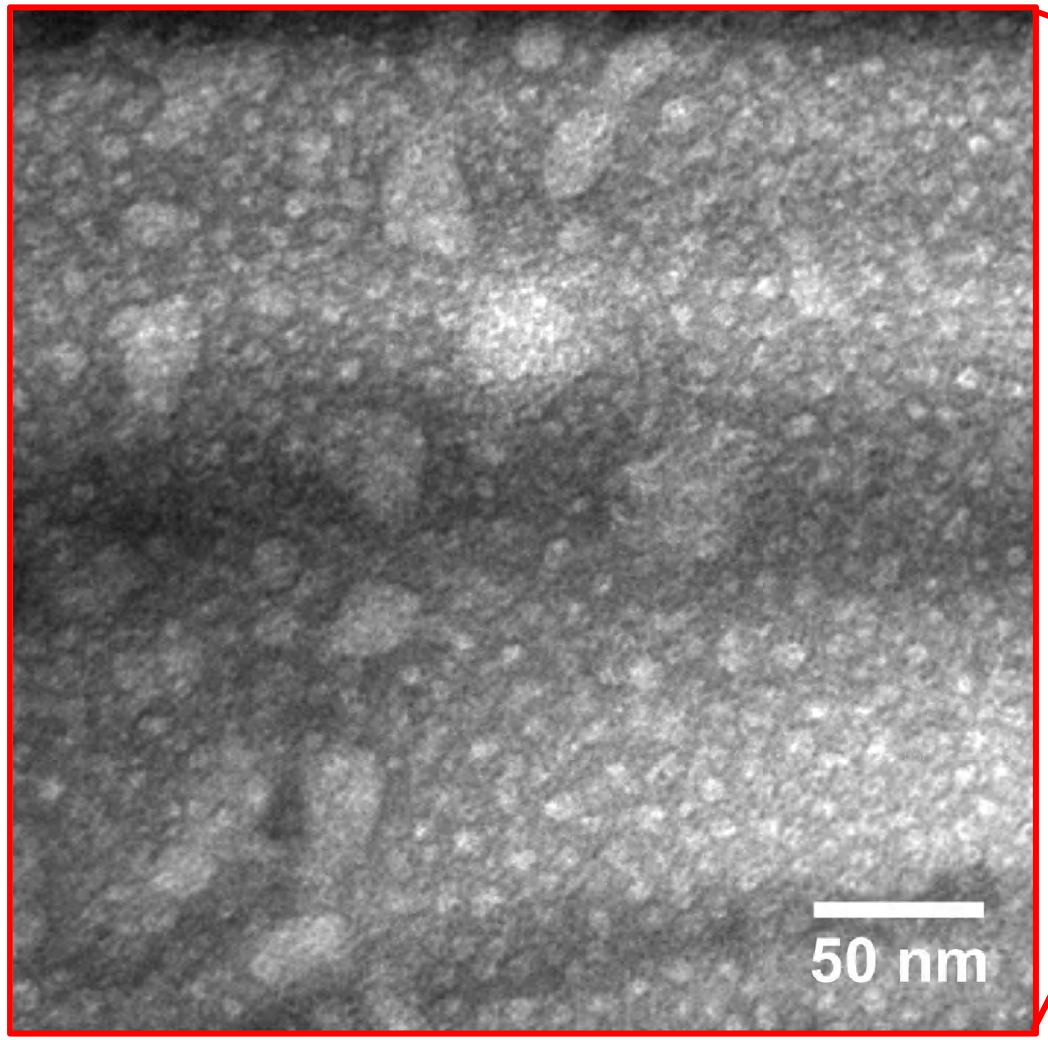
3×10^{23} H/m^2 (415 eV/ion, 300 K)



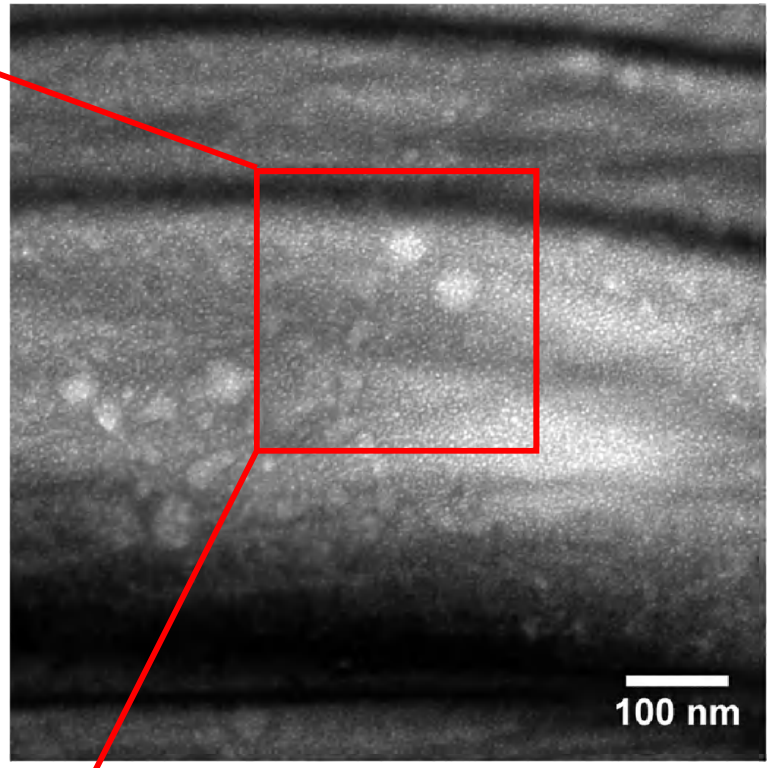
- Specimen overview.

HSSL: defect microstructure by TEM

3×10^{23} D/m^2 (415 eV/ion, 300 K)



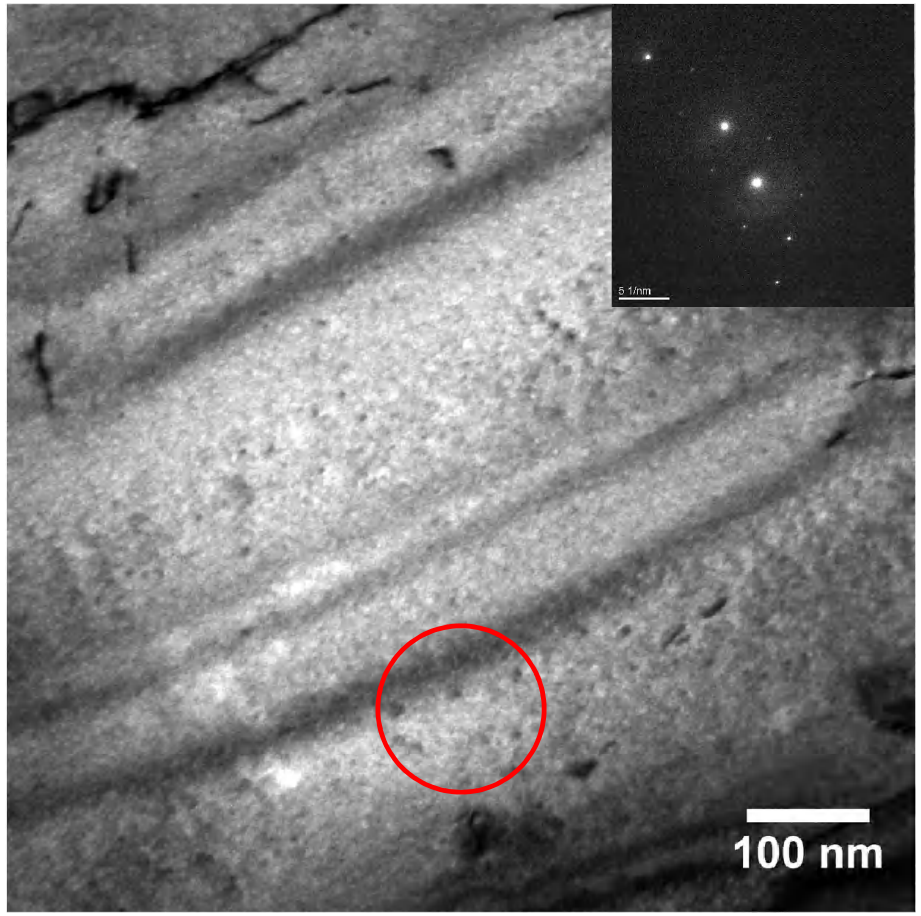
Thin region



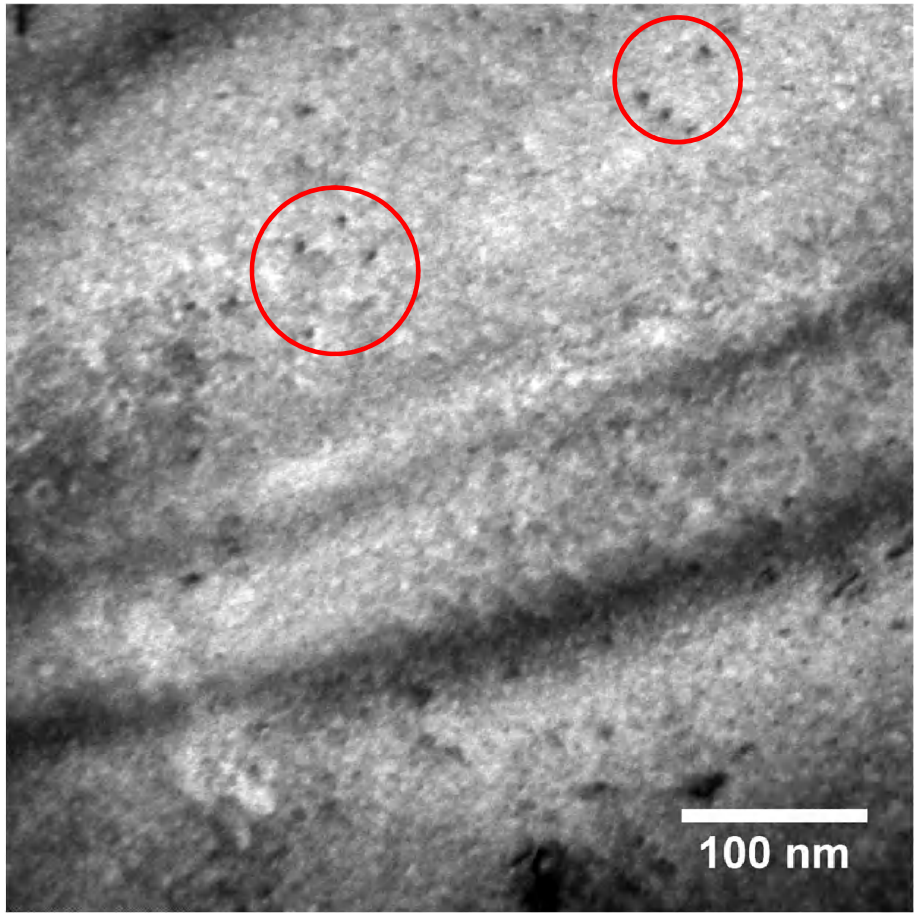
- Electron beam exposure/< 2 min

HSSL: defect microstructure by TEM

3×10^{23} H/m^2 (415 eV/ion, 300 K)



Thin region

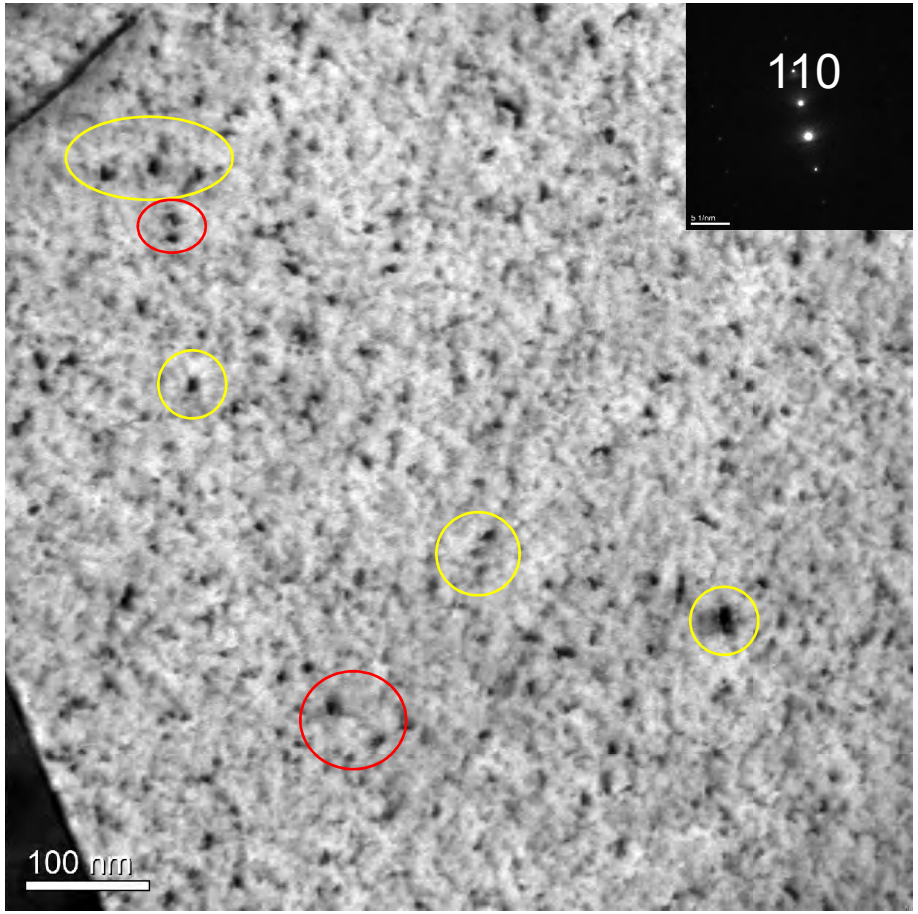


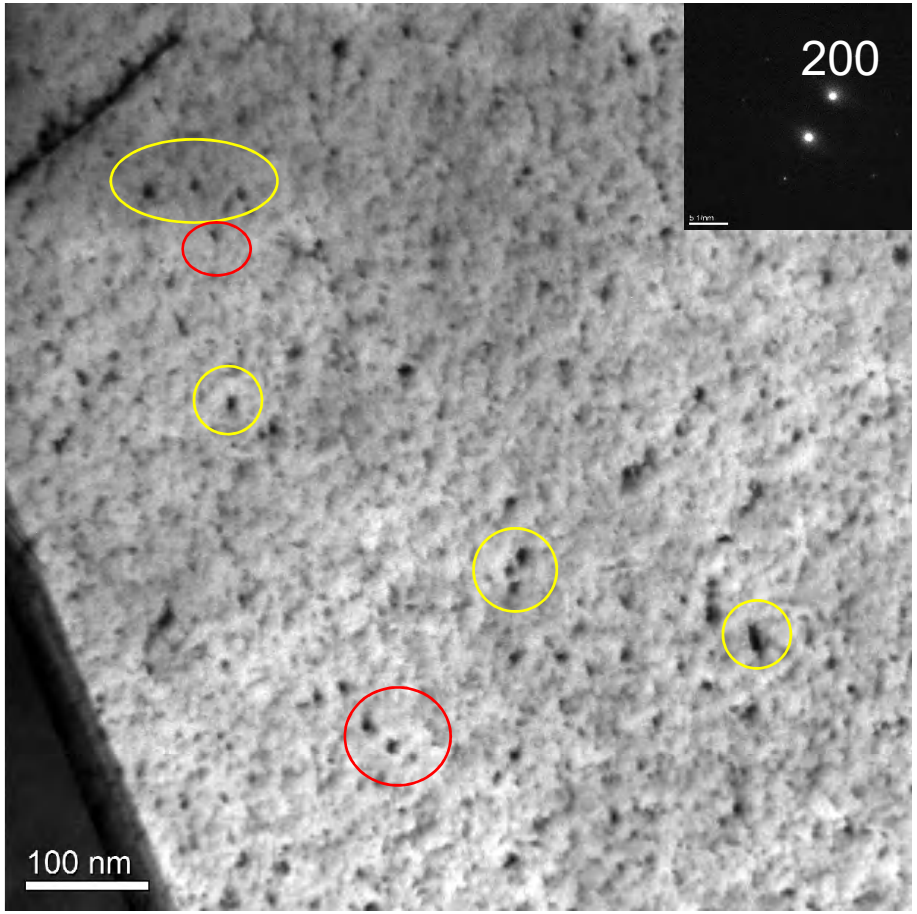
- Electron beam exposure < 2 min
- Production of black-spot contrast damage

HSSL: defect microstructure by TEM

6×10^{23} H/m^2 (415 eV/ion, 300 K)

Thick region (100-150 nm)



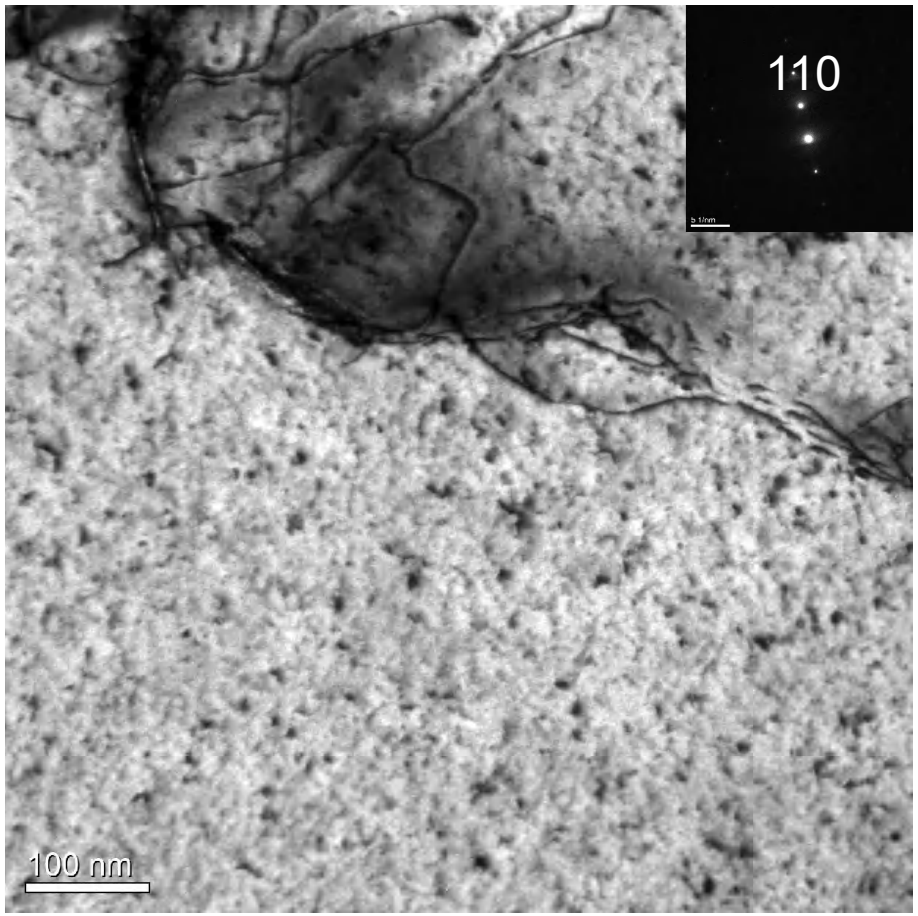


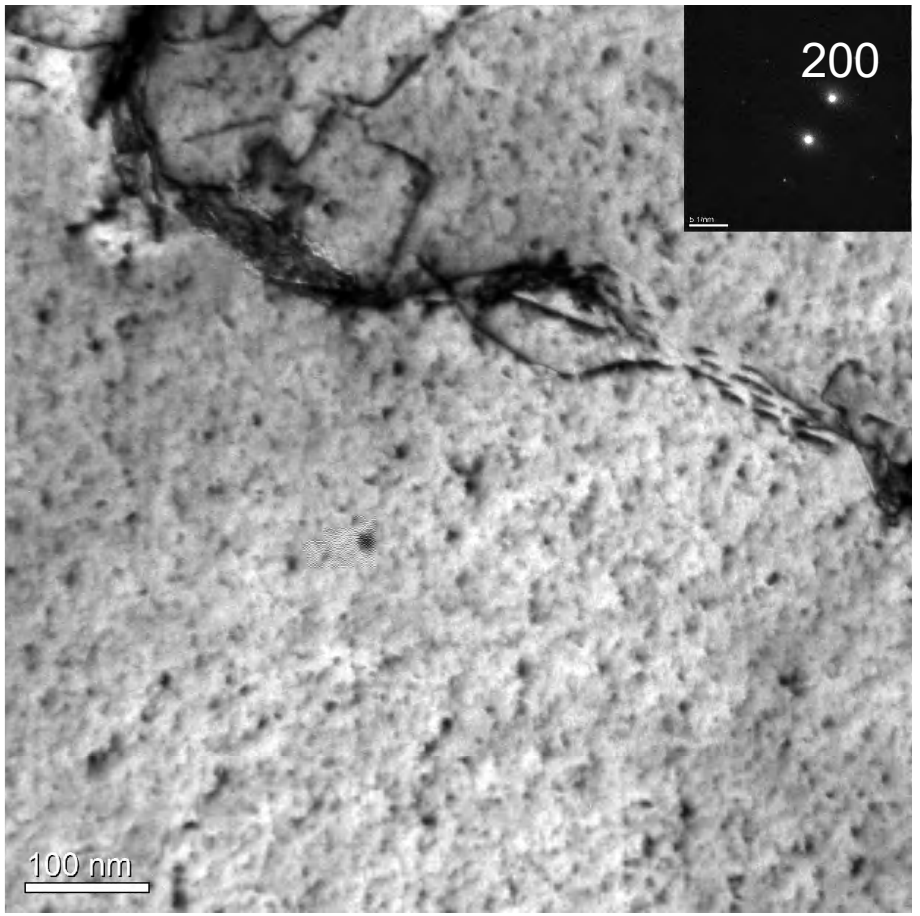
- Black spot contrast damage: max. size <15 nm ; density $(7.2\pm0.6)\times10^{21}$ m^{-3};
- g.b criterion applies.

HSSL: defect microstructure by TEM

6×10^{23} H/m^2 (415 eV/ion, 300 K)

Thick region (100-150 nm)





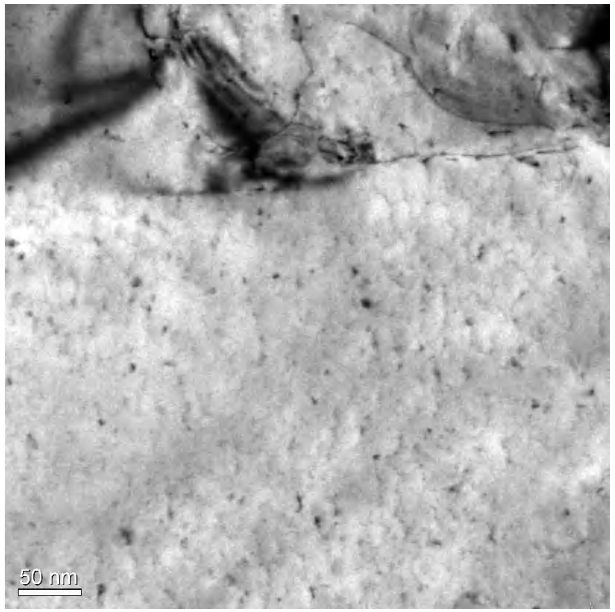
- Black spot contrast damage versus dislocation networks introduced during thermo-mechanical working.

HSSL: defect microstructure by TEM

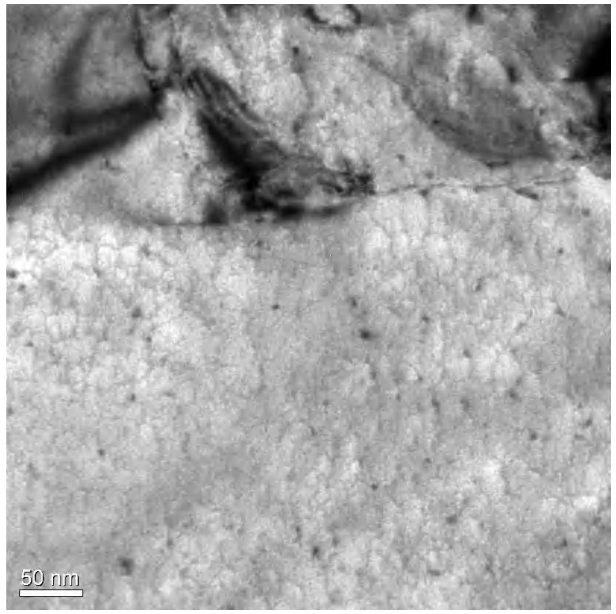
6×10^{23} H/m^2 (415 eV/ion, 300 K)

Thin region

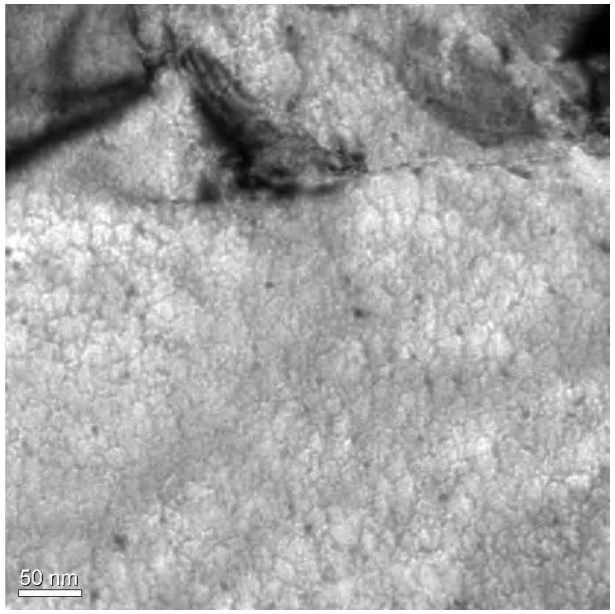
Δf = 0



Δf = -2 µm



Δf = -4 µm

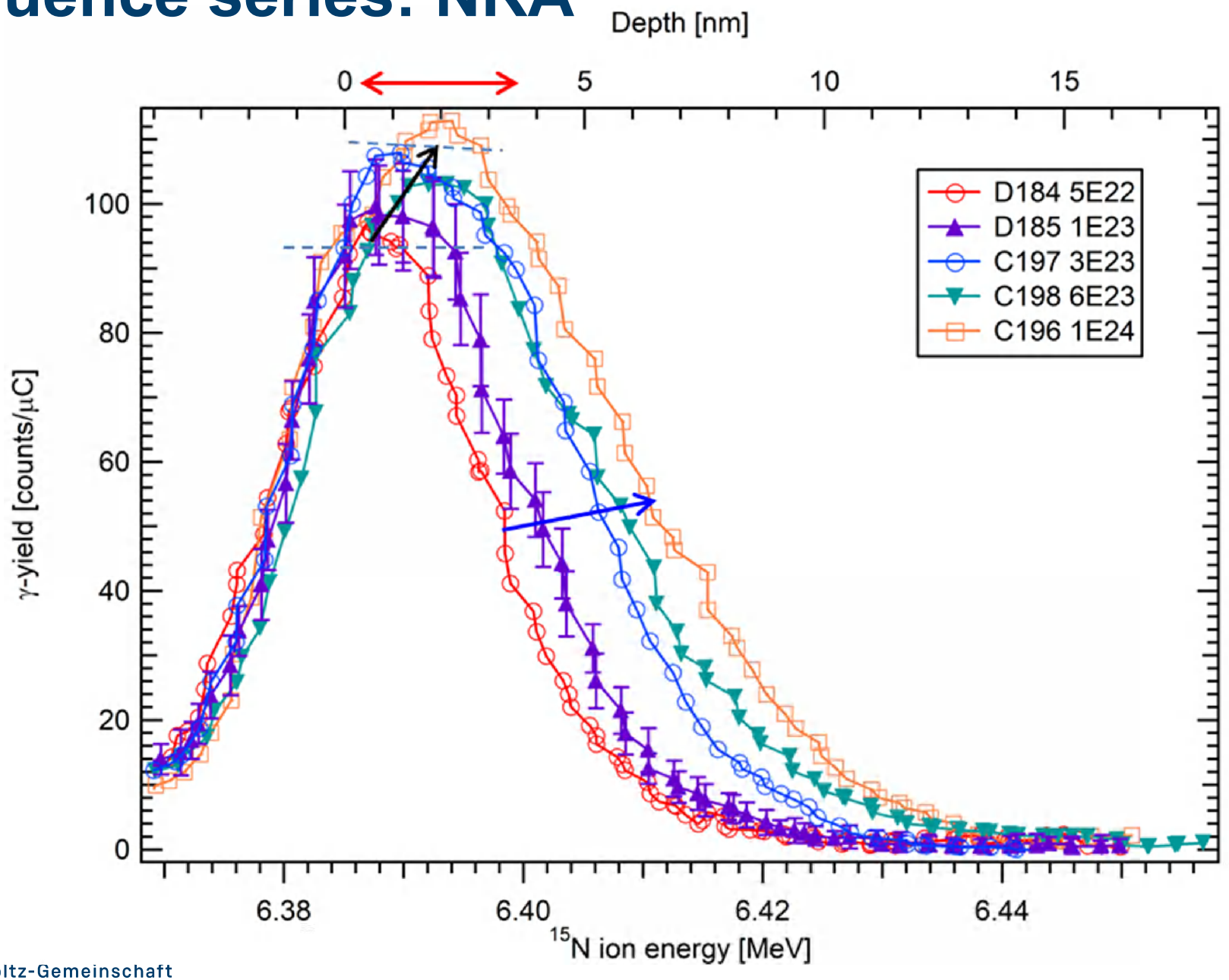


- Presence of dense/overlapped bubbles.
- Bubble max. size < 40 nm, density $(3.6\pm0.4)\times10^{22}$ m^{-3}.
- Bubbles remained stable under 200 keV electron beam exposure up to 10 min, no dynamic behaviour observed.

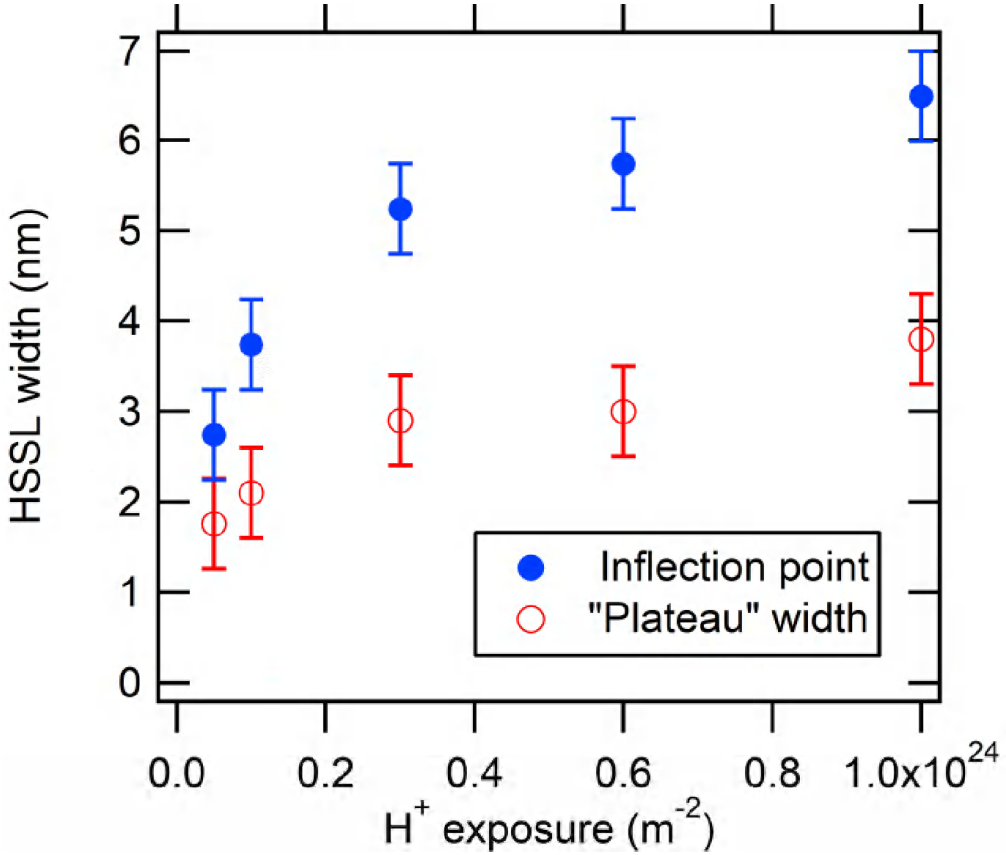
TEM characterization of SSL: chapter summary

- ❑ First observation of spherical nanovoids by H plasma exposure
- ❑ Clear defects evolution are observed: nanovoids, black spots…
- ❑ Further TEM characterization is ongoing…

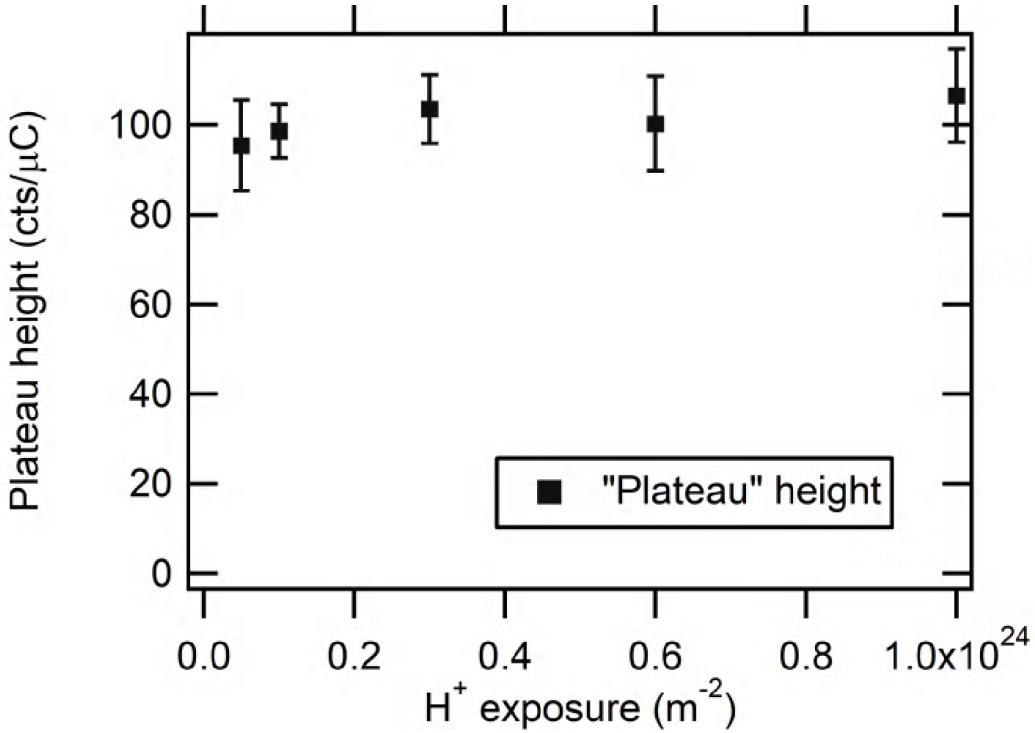
HSSL fluence series: NRA



HSSL fluence series: NRA



- **HSSL "plateau" height** (~100 ± 10 cts/μC): **8-9 at. % H**
- **Inflection point** (2.7 -> 6.5 nm) → **HSSL thickness**)



H/D-SSL: take-home message

- ❑ 10 at.% HI in SSL is reached already at 5e22 H/m^2 (rather low: a few minutes in low-flux (10^{19} – 10^{20}) plasma source).
- ❑ SSL increases only in thickness but not in H concentration with exposure fluence: saturation
- ❑ TEM-visible defects evolve but HI retention stays constant: further characterization is required.

Thank you for your attention!