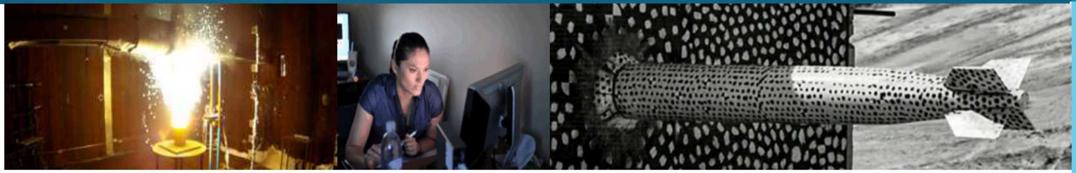
SAND2020-1878C

The critical microstructural conditions for void nucleation during ductile rupture: dislocation structures and vacancy condensation



Philip J. Noell, Julian E. C. Sabisch, Douglas L. Medlin, Jay D. Carroll, Brad L. Boyce

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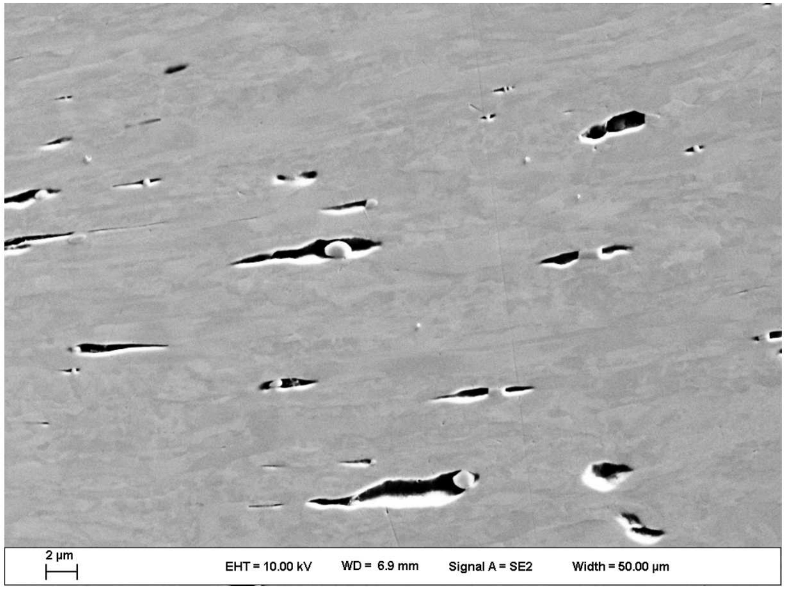
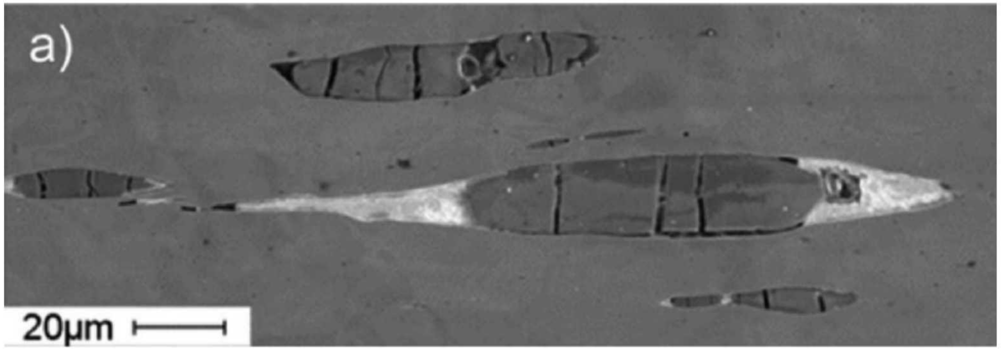
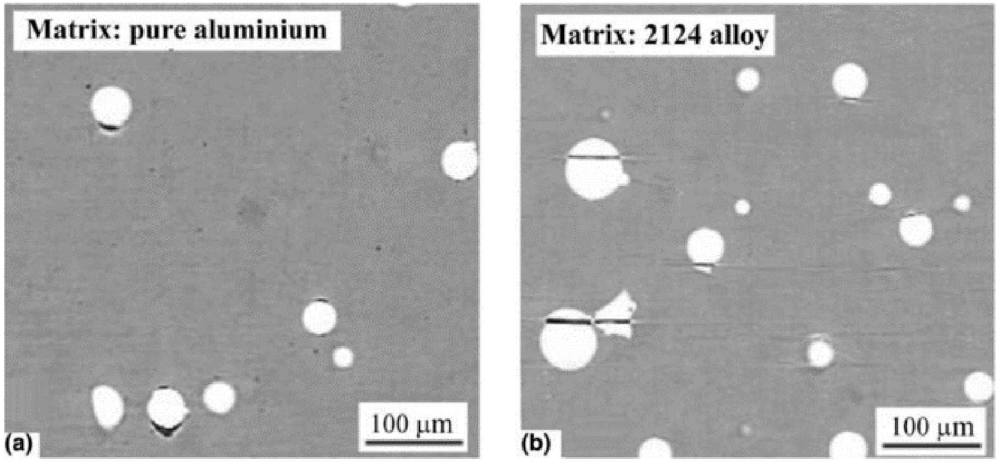
Void nucleation during ductile rupture



Secondary-electron image of voids in a deformed copper wire containing second-phase particles



Cracked MnS inclusions in a free-cutting steel. Seo et al, ISIJ International, 2015.



Babout, L. *et al*, *Acta Mater.*, (2004)

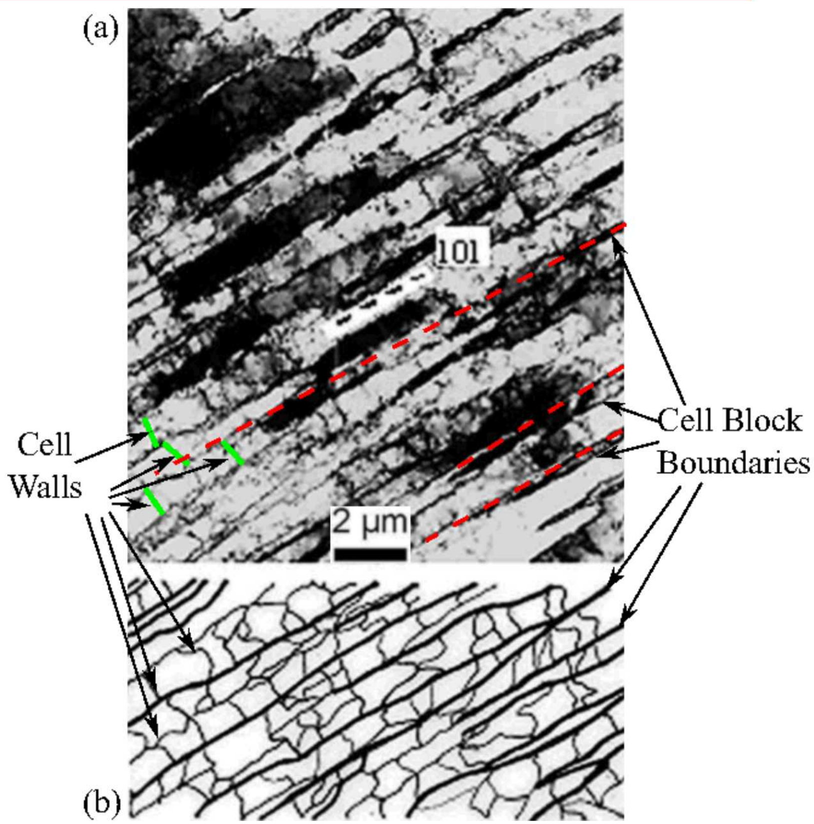
Void nucleation in many materials is intragranular and associated with inclusions and/or second-phase particles, either by particle fracture or by particle decohesion.

Does microstructural evolution in the matrix during deformation play a role?

Deformation-induced defect structures

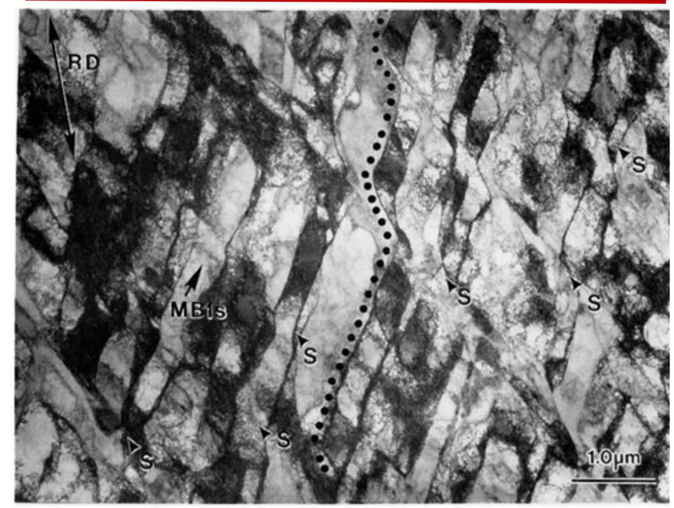
- Deformation twins
- Slip bands and dislocation pileups
- Dislocation boundaries
    - cell block boundaries, cell walls, microshear bands
- Dislocation loops, stacking fault tetrahedra
- Vacancy clusters

Cell blocks and cell walls in deformed iron



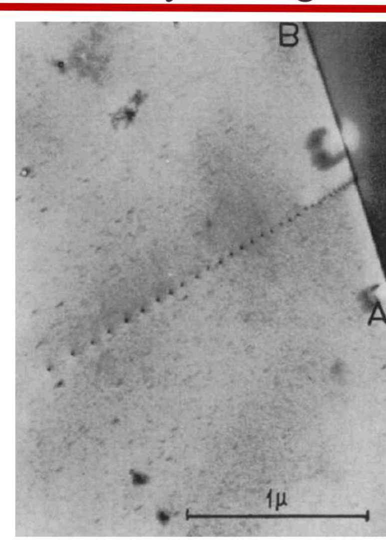
Li, B. L., *et al. Acta Materialia* 52.4 (2004)

A microshear band cutting through cell blocks in Ni



Hughes, D.A. *et al., Met Trans A,* 1993

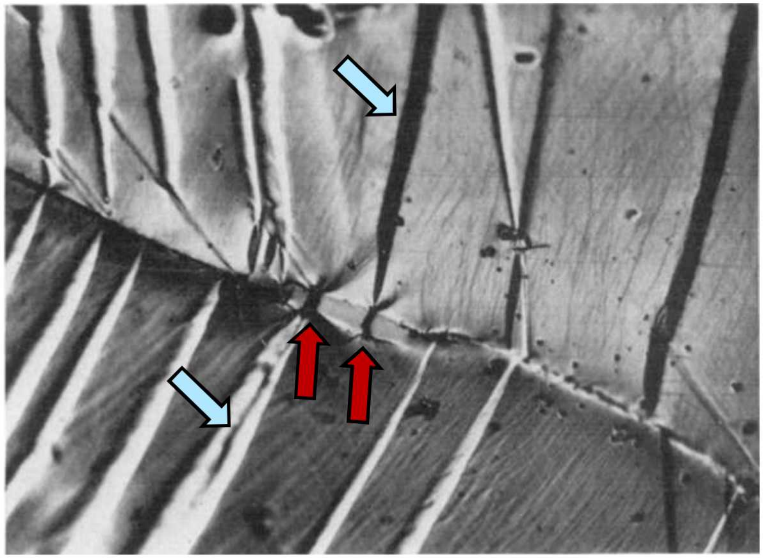
A dislocation pile-up at a grain boundary in tungsten



Wronski, A. *et al.*, J. Less Comm. Metl, 1964

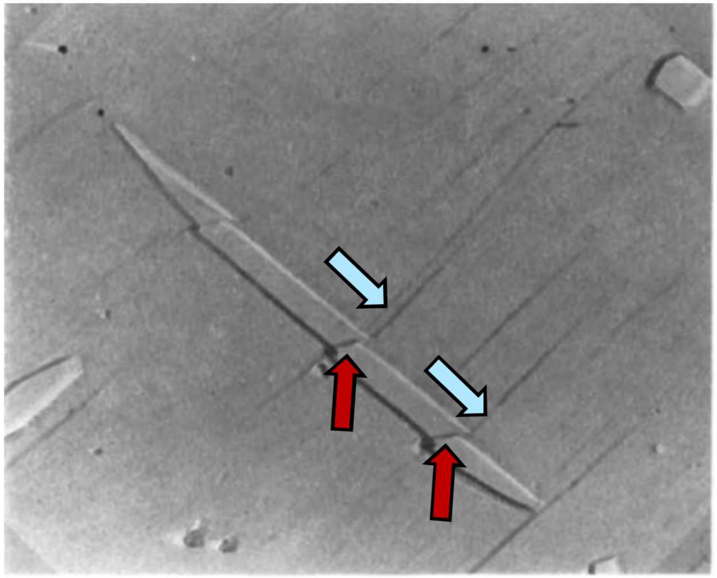
Relating particle-stimulated void nucleation to deformation-induced defect structures: dislocation pileups and deformation twins

Fractured hydride platelet in Zr associated with $\{11\bar{2}\bar{1}\}$ type twins; **particle fracture depended on twin type**



Warren, M. *et al., J. Nuc. Mat.*, 1968

A cracked carbide associated with slip bands in an austenitic stainless steel



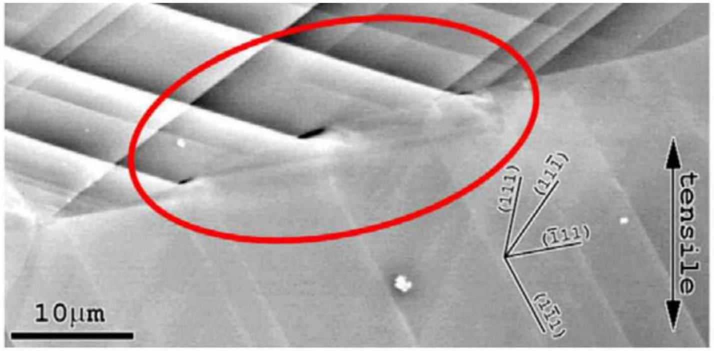
Barnby, J., *Acta Metallurgica*, 1967

- Early studies of void nucleation based on secondary electron images, overlooked the role of dislocation boundaries in void nucleation

- Modern X-ray tomography studies of void nucleation no insight into relationship between void nucleation and defect structures

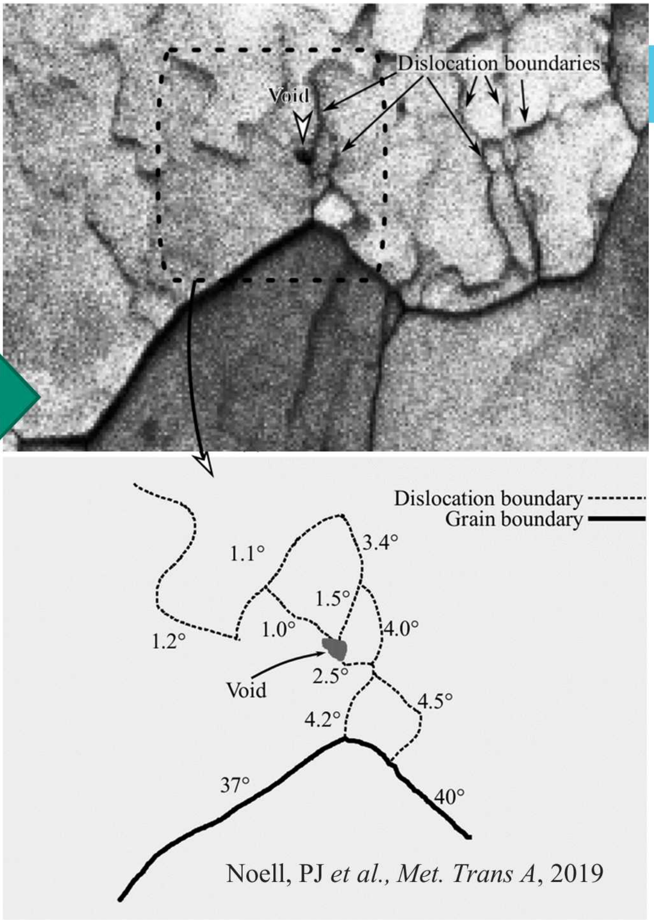
The microstructural features associated with particle-free void nucleation in bulk materials

Microcracks in TiAl correlated with twin intersections.



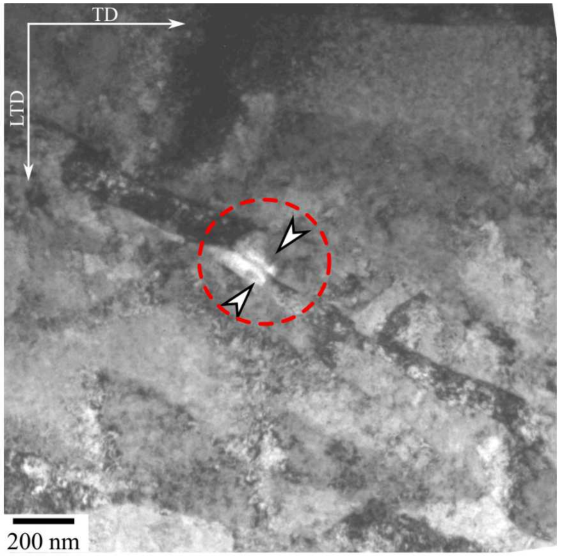
Bieler, T. R., *et al., International Journal of Plasticity,* 2009

Void nucleation in Al occurred at dislocation cell walls, suppressed by DRX

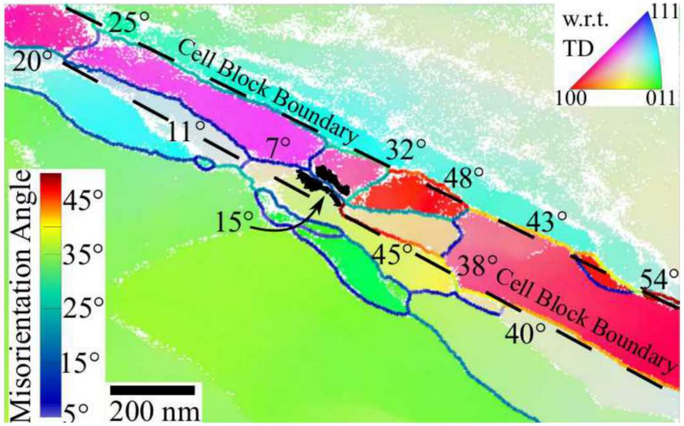


Noell, PJ *et al., Met. Trans A*, 2019

- **In bulk, pure metals that deform by twinning, voids nucleate at grain-boundary/twin intersections and twin/twin intersections**
- **In bulk pure metals that deform by slip, voids nucleate at dislocation boundaries**



Void nucleation in Ta primarily occurred at cell block boundaries

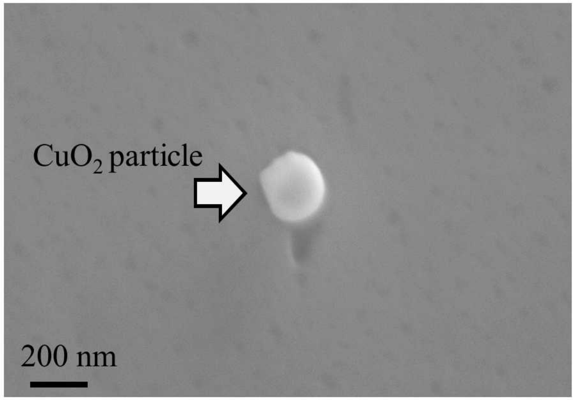


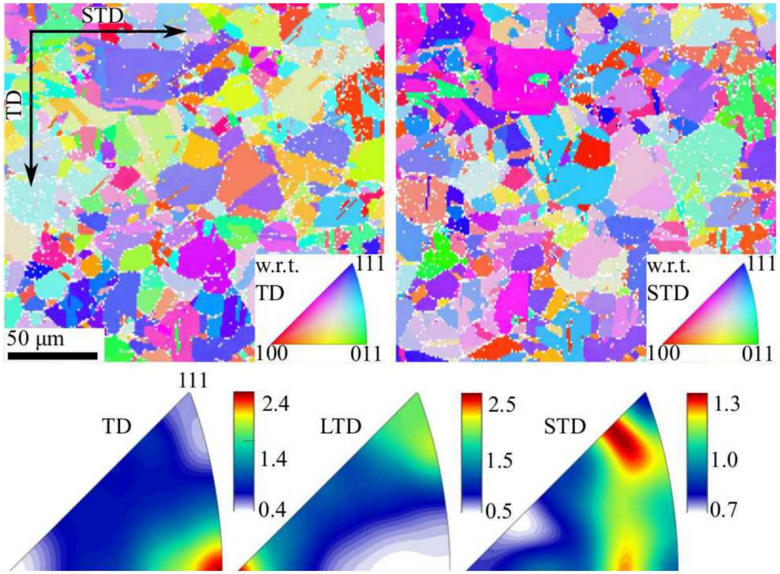
Noell, PJ et al., *Acta Mat,* 2017

Do deformation-induced defect structures also enable the formation of microscale voids in materials that contain second-phase particles?

Material: 99.9% Cu sheet material, annealed after rolling

Contains ≈200 to 500 nm copper oxide inclusions, ≈1 inclusion per 400 μm² of material

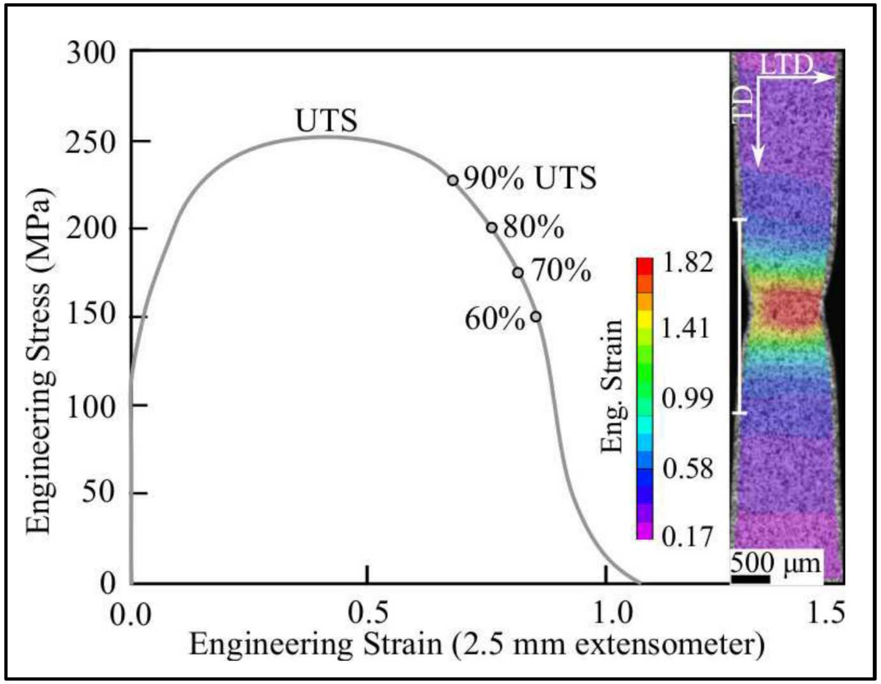




EBSD indicates that the as-received microstructure contained weakly-textured, equiaxed grains (8.7 μm diameter)

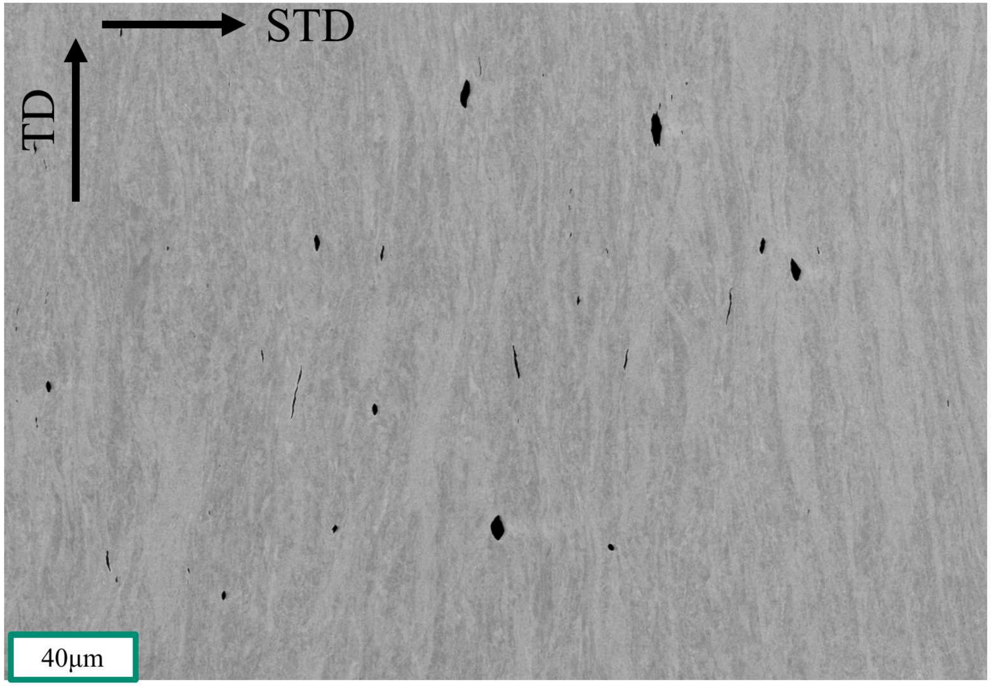
1. What deformation-induced defect structures are associated with the formation of microscale voids?
2. Are copper oxide inclusions the primary nucleation site for voids in this material?

Incipient, microscale voids first appear at 70% UTS, observed throughout diffuse neck by 60% UTS



To understand the origins of these voids, interrupted tensile tests used to produce specimens containing incipient voids

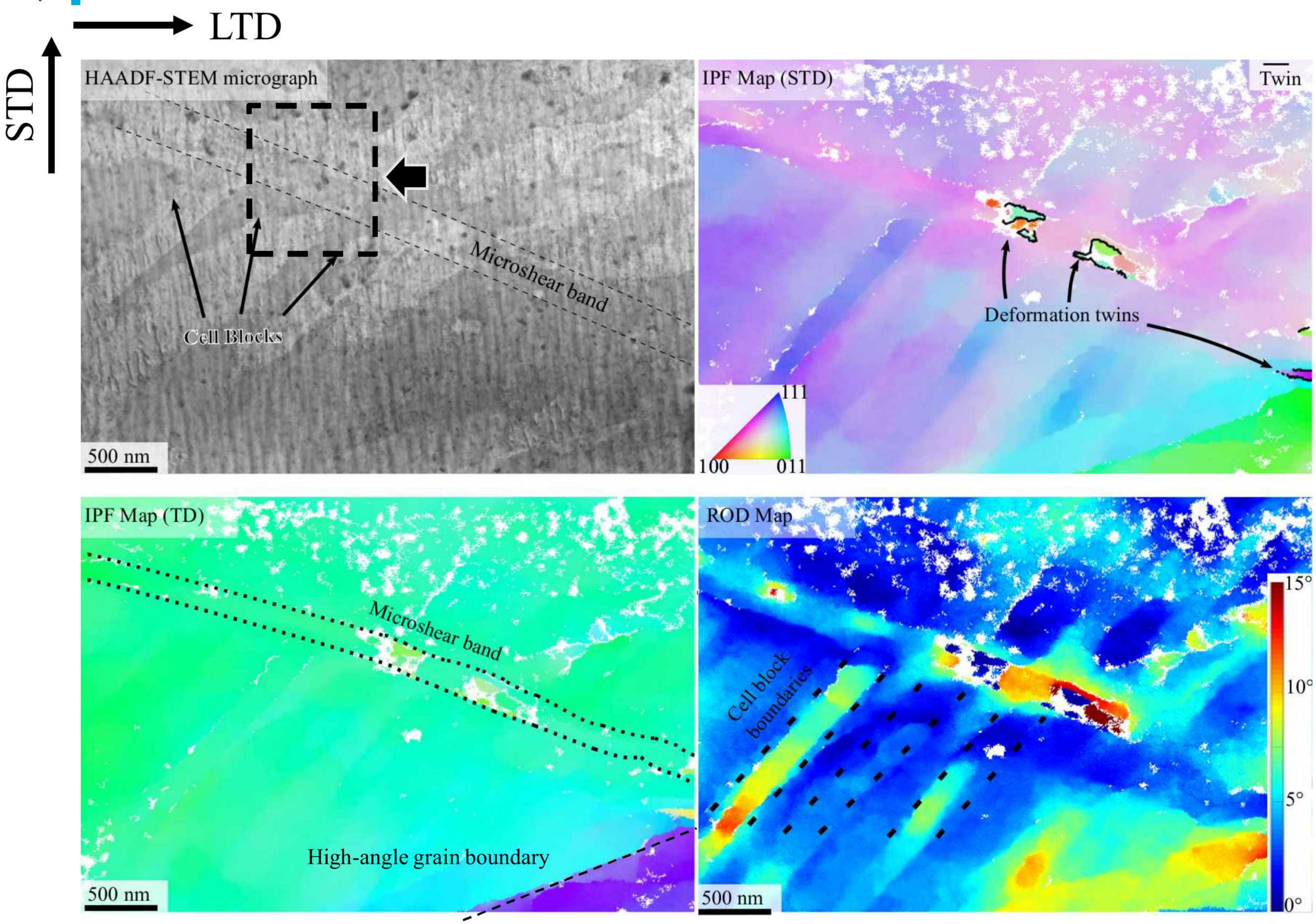
- Area density of microscale voids in the center of the neck of a 60% UTS sample was ≈ 1 per 400 μm^2.
- Voids were oblong (elongated along TD), largest dimension ranged from ≈0.5 to 10 µm



Incipient voids at 60% UTS

- ❑ **Defect structures in the deformed material**
- ❑ The origins of microscale voids

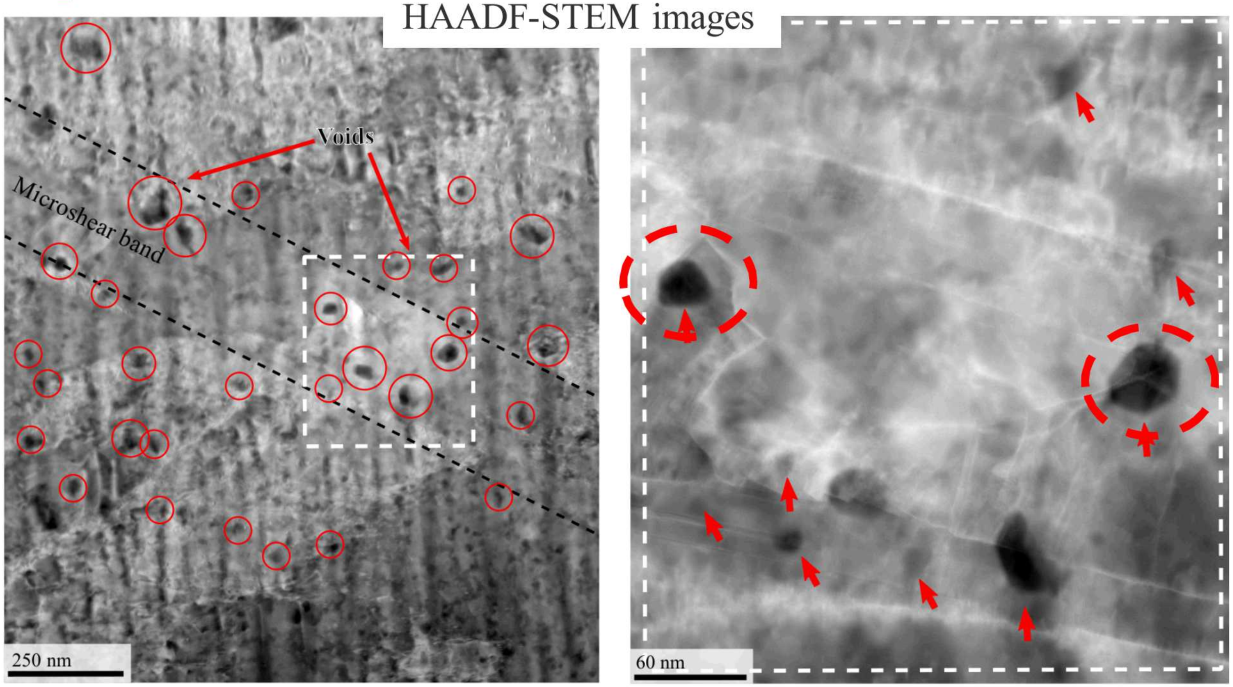
Deformation-induced defect structures



Deformation twins and microshear bands observed at the microscale

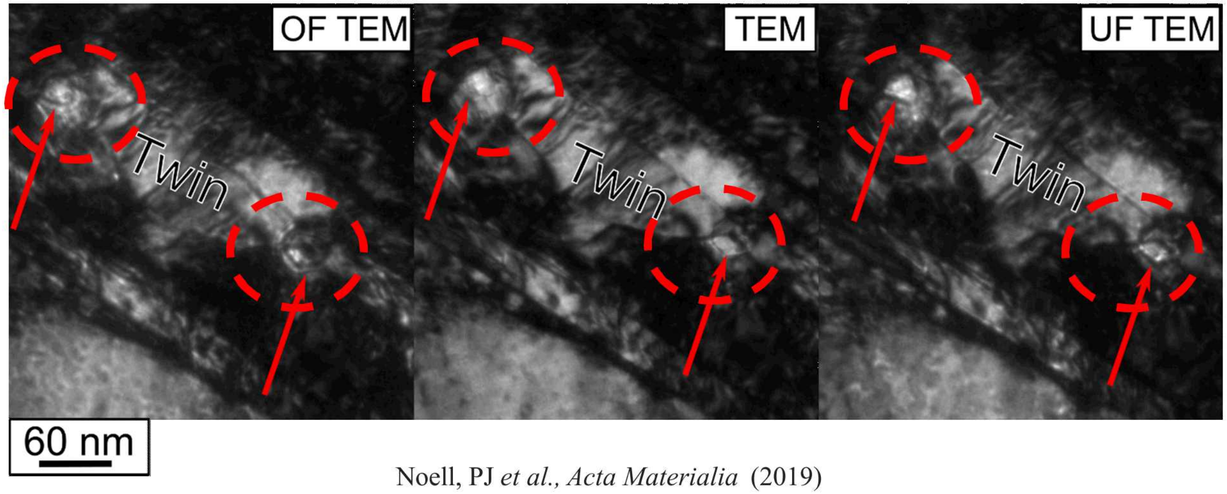
Noell, PJ *et al.*, *Acta Materialia* (2019)

Vacancy clusters/nanoscale voids



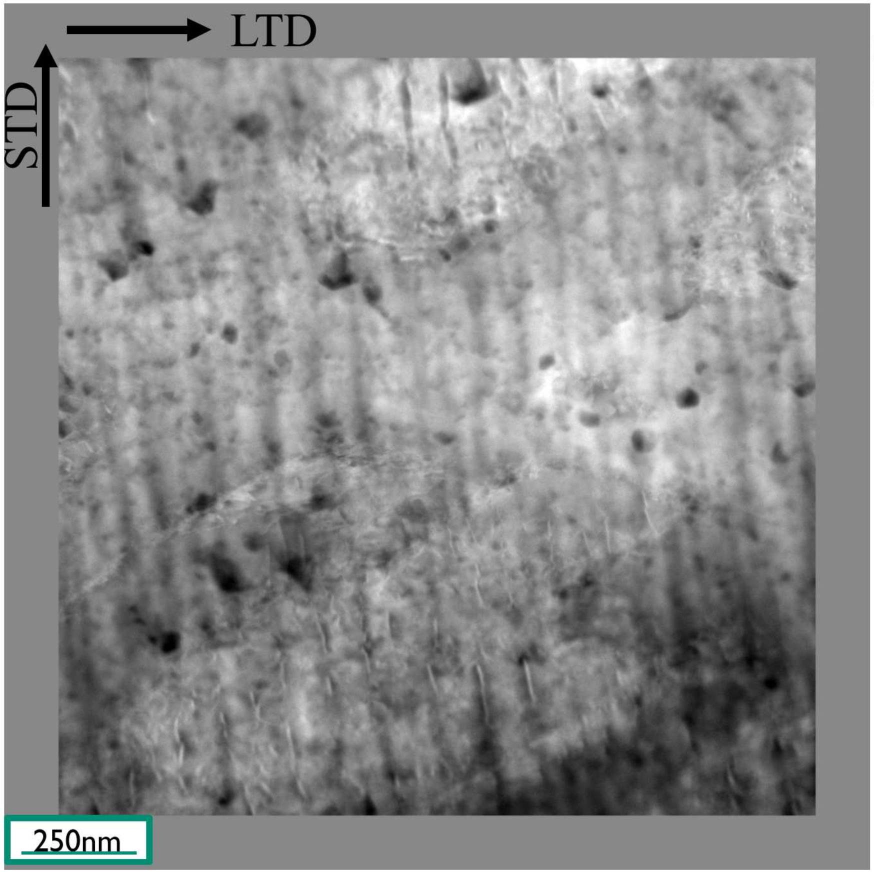
- Dozens of faceted, nanoscale (≈10-100 nm) voids observed both within and outside the microshear band
- ***No evidence in EDS or diffraction measurements that suggests a change in composition or secondary-phase***

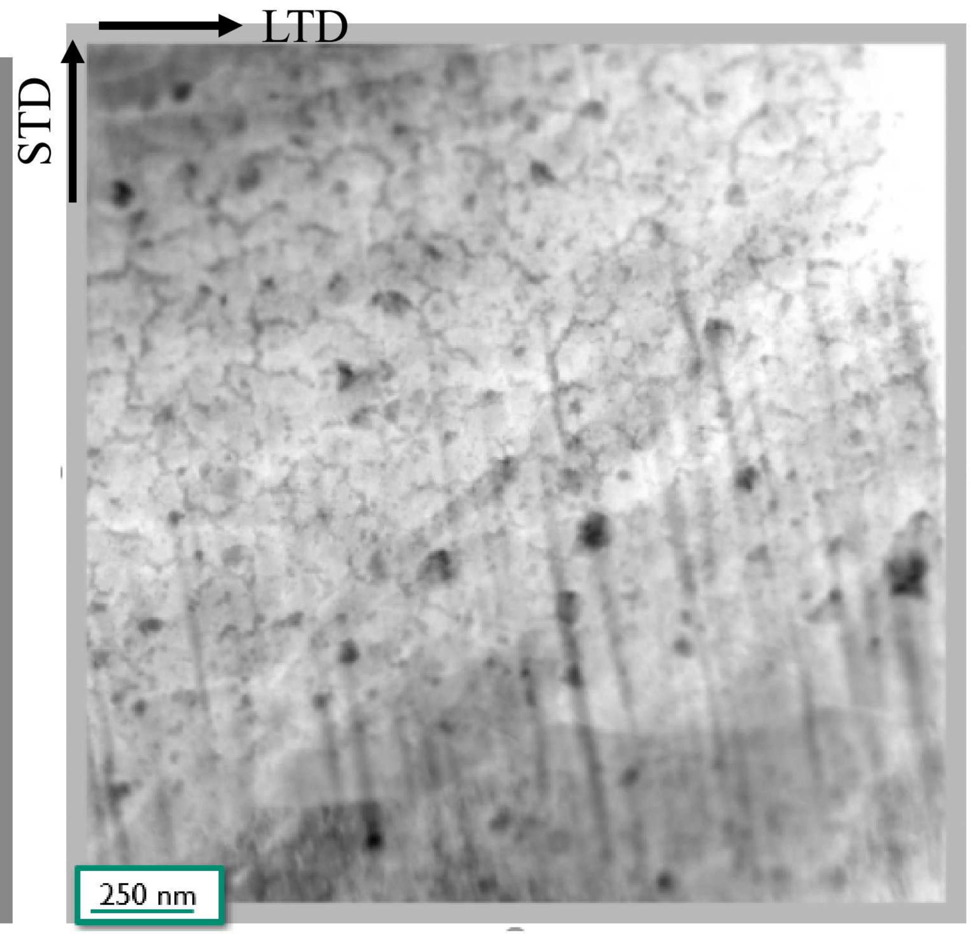
- Phase-contrast TEM (overfocus (OF) and underfocus (UF) images are provided) highlights changes in Fresnel contrast through the focus series within the highlighted areas that are consistent with a reduction in local density as expected for voids



Noell, PJ *et al., Acta Materialia* (2019)

A high density of nanoscale voids appear to be distributed throughout the midplane of the diffuse neck





- Estimate from micrographs suggests a density of nanoscale voids of 10^2 per um^2
- Observed both within microshear band and outside, suggesting they are distributed homogenously throughout necked region

The origins of nanoscale void nuclei: vacancy condensation

Proposed void nucleation mechanisms include:

- Particle decohesion
- Particle delamination
- Grain boundary cleavage
- Vacancy condensation

Void nuclei will be distributed heterogeneously at specific microstructural features

Void nuclei will be fairly homogeneous

$$c_v = \frac{A}{G}\int_0^{\varepsilon} \sigma d\varepsilon$$

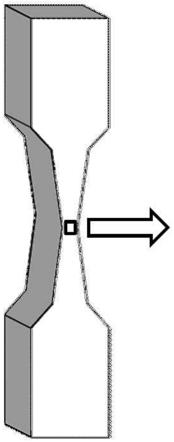
C_v - vacancy concentration
A - constant (A~0.1)
G - shear modulus

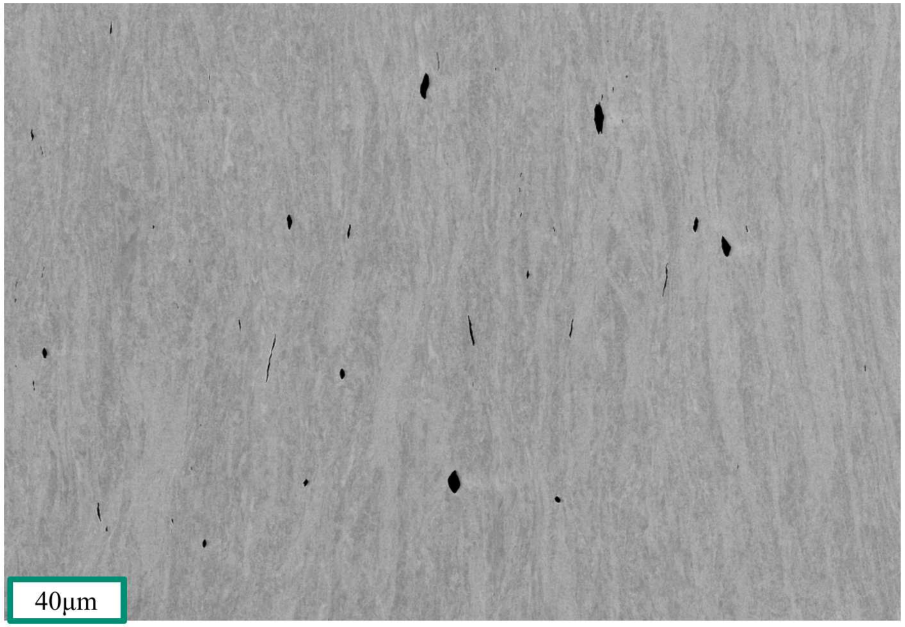
- Vacancy supersaturation in Cu specimens at $\varepsilon = 60\%$ experimentally measured to be between 10^{-7} and 10^{-4} (T. Ungar, *et al.*, *Mat Sci Eng A*, (2007))
- Vacancy concentration in neck at $\varepsilon \approx 150\%$ predicted to be $\approx 10^{-4}$

- Void nucleation in this material is **not** controlled by inclusions or second-phase particles
- This result strongly suggests that vacancy condensation contributes to void nucleation

- Defect structures in the deformed material
- **The origins of microscale voids**

What microstructural features are associated with the handful of voids that grow?



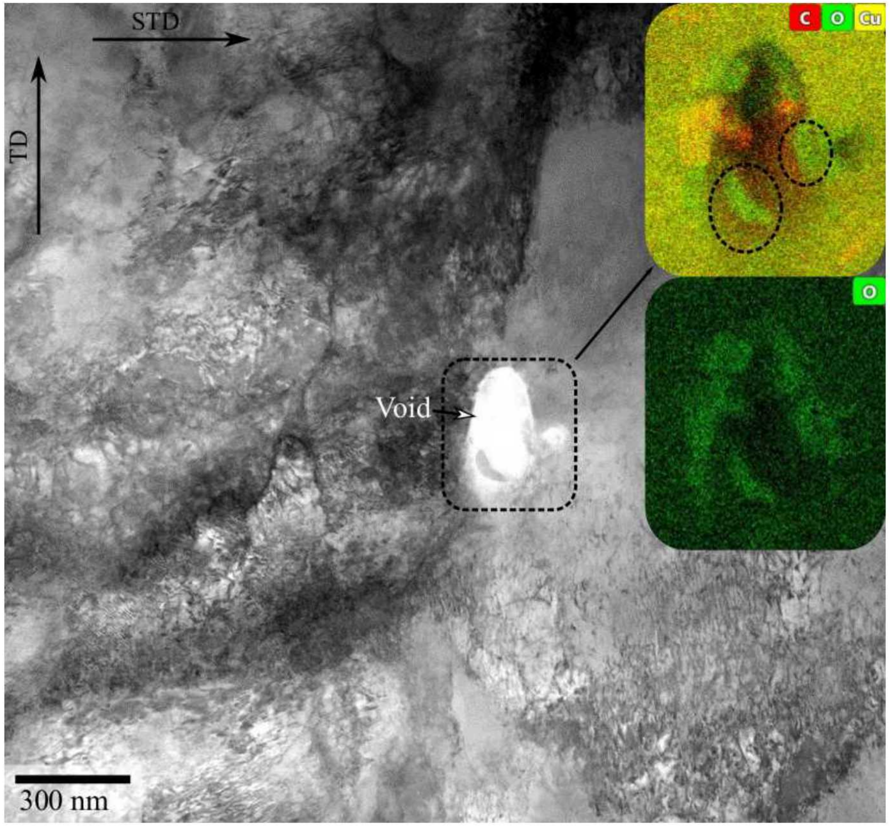


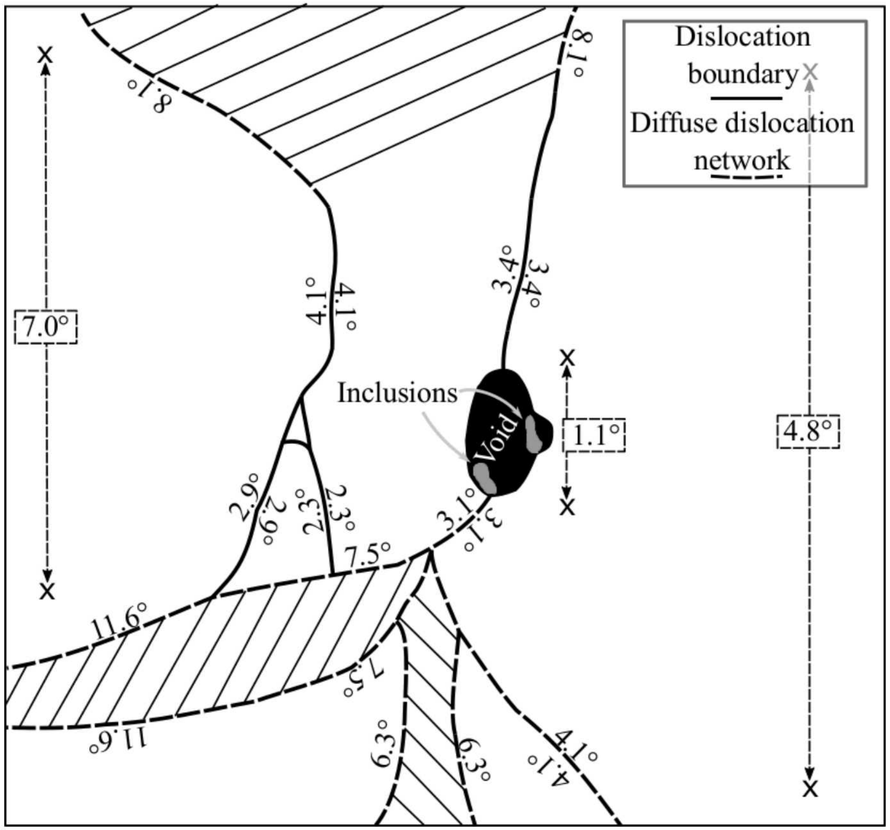
Conservatively, only 1 in 10,000 nanoscale voids grows to the microscale. ***Void nuclei thus appear to be overseeded!***

22 incipient (< 2 μm) voids in the diffuse necks of 70% and 60% UTS specimens characterized using a combination of EBSD (20) and TEM (2)

- **All** voids associated with a deformation-induced dislocation boundary
- 13 were particle free and emerged within a grain
- 6 emerged at the inclusion-free intersection between a grain boundary and one or more dislocation boundaries
- 5 emerged at an inclusion that was intersected by a dislocation boundary and, in some cases, a grain boundary

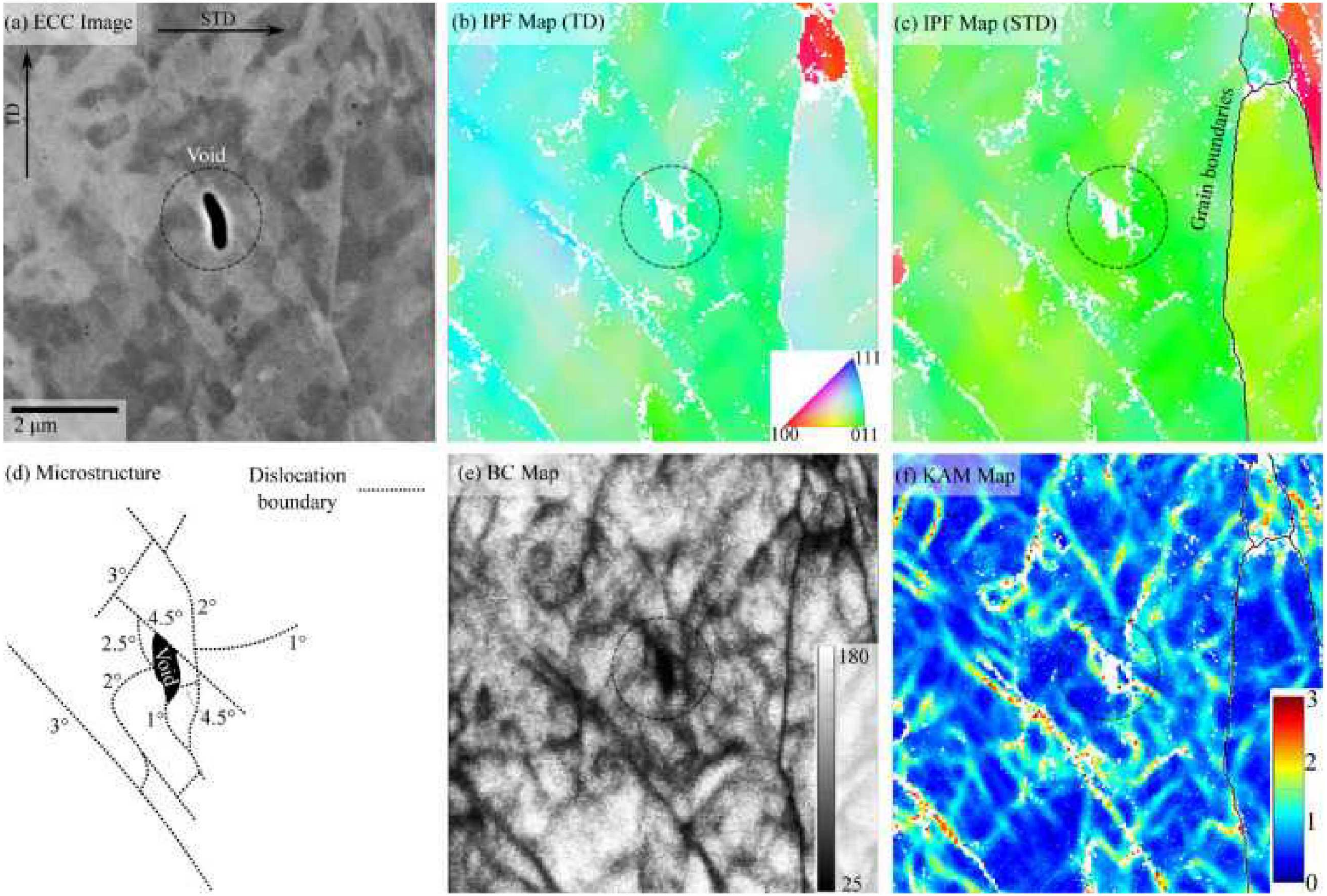
Particle-stimulated voids: nanoscale





Even at the nanoscale, it is clear that dislocation boundaries are associated with the emergence of voids that are associated with particles

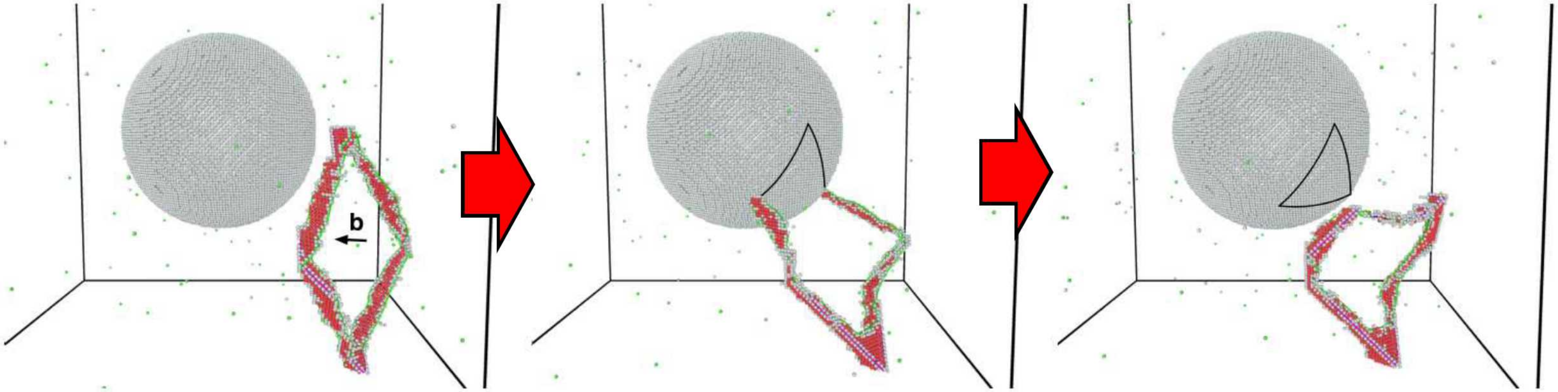
Intragranular, inclusion-free voids



Most microscale voids emerged within grains and were not associated with inclusions or particles

These observations suggest void emergence to the microscale depends on dislocation structures

Sills, R. *et al., MRL* (2019)



(a) Dislocation line attracted to void

(b) Dislocation glides across void, cross-slips onto another glide plane

(c) Process repeated until dislocation glides free. Result is a closed dislocation loop traced out on the void's surface, adding to its volume.

- MD simulations of void growth by dislocation absorption suggest that this is a realistic mechanism for the growth of nanoscale voids
- This mechanism is only realistic if the nanovoid is surrounded by a high-density of mobile dislocations, which is expected if the nanovoid is intersected by one or more dislocation boundaries

Conclusions

1. What deformation-induced defect structures are associated with the formation of microscale voids?
2. Are copper oxide inclusions the primary nucleation site for voids in this material?

- Voids nucleated at the nanoscale, perhaps by vacancy condensation, and were distributed fairly homogeneously within the midplane of the necked gauge region
- ***Void nuclei appear to be overseeded.*** The formation of microscale voids thus controlled by the emergence of nanoscale voids to the microscale (early stages of void growth) and ***not*** by void nucleation.
- The early stages of growth depend primarily on the location of the void nuclei and appears to be controlled by the dislocation structures that intersect the void.
- ***The evolution of dislocation structures appears to control the formation of failure-critical voids.***