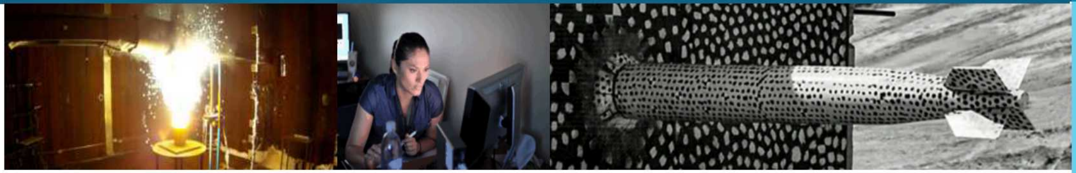
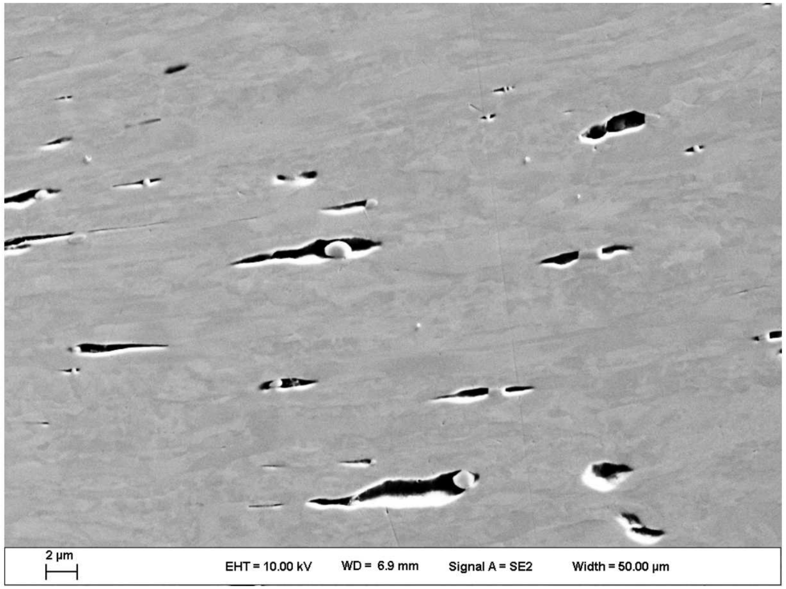
SAND2020-0016C

The Critical Microstructural Conditions Leading to the Formation of Voids During Ductile Rupture

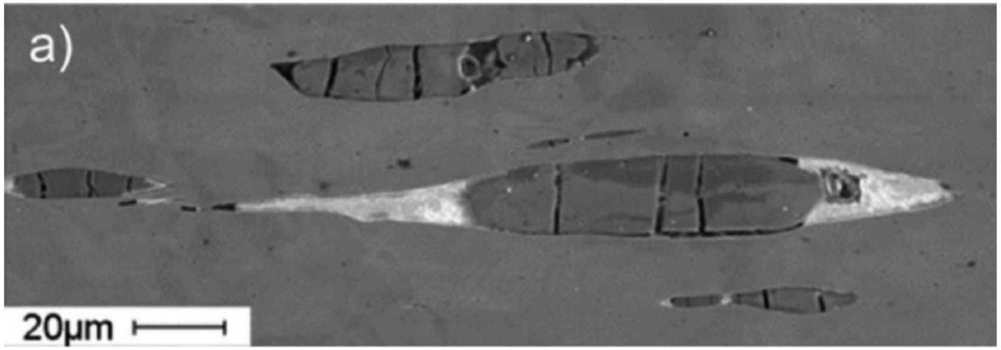


Philip J. Noell, Julian E. C. Sabisch, Douglas L. Medlin, Jay D. Carroll, Brad L. Boyce

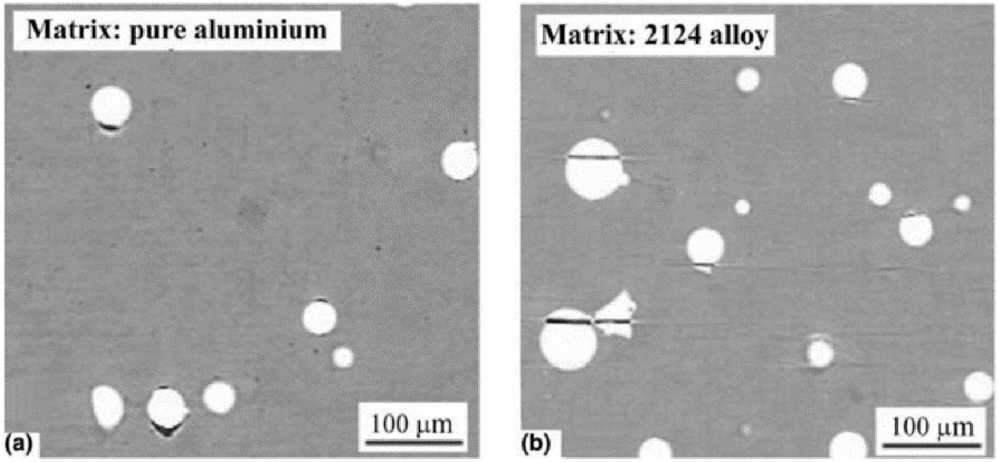
Void nucleation during ductile rupture



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)

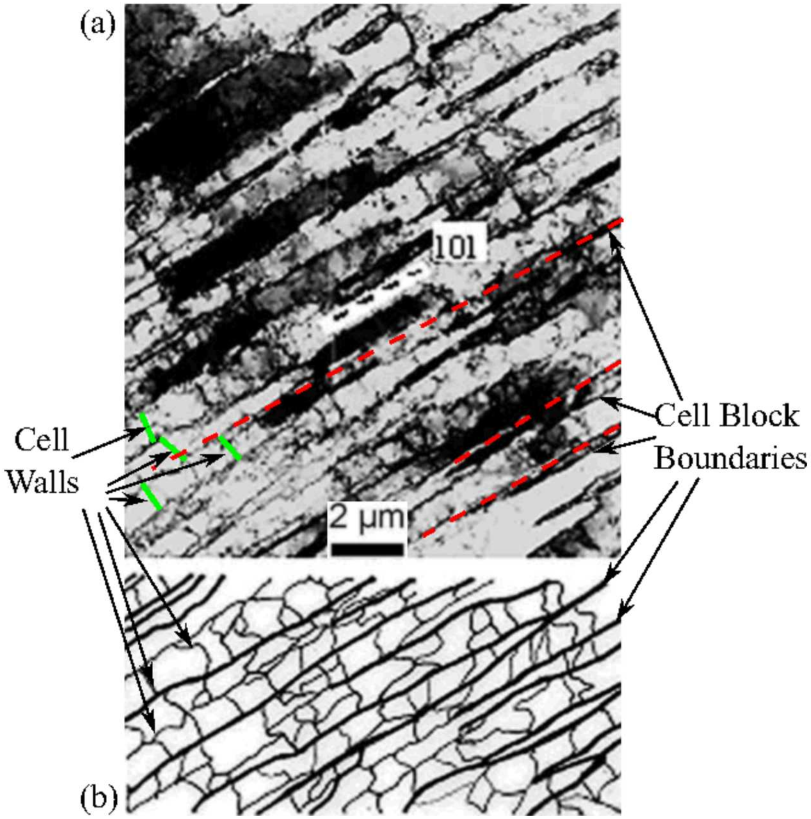
Void nucleation in many materials is intragranular and associated with inclusions and/or second-phase particles, either by particle fracture or by particle decohesion.

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

Deformation-induced defect structures

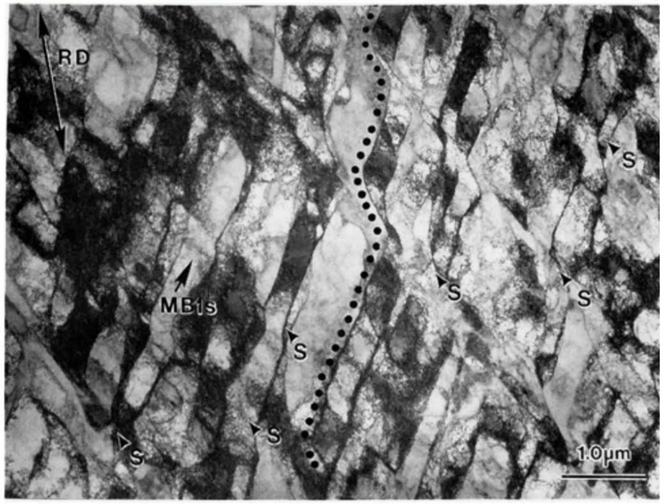
- Deformation twins
- Slip bands and dislocation pileups
- Dislocation boundaries
  - cell block boundaries, cell walls, microshear bands
- Dislocation loops, stacking fault tetrahedra
- Vacancy clusters

Cell blocks and cell walls in deformed iron



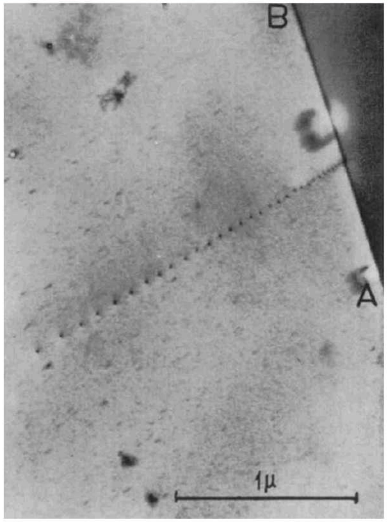
Li, B. L., *et al. Acta Materialia* 52.4 (2004)

A microshear band cutting through cell blocks in Ni



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

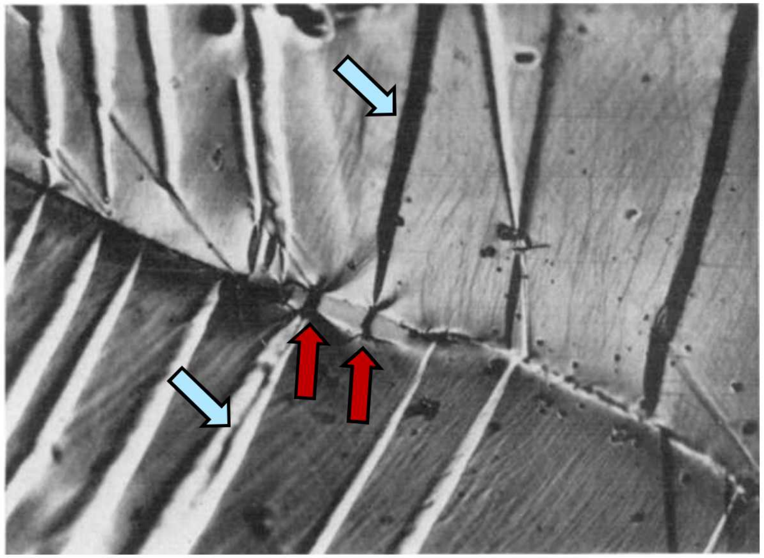
A dislocation pile-up at a grain boundary in tungsten



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

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

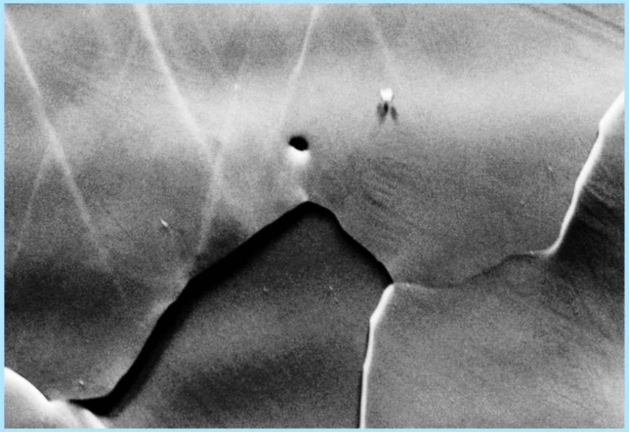
Fractured hydride platelet in Zr associated with $\{11\overline{2}\overline{1}\}$ type twins; **particle fracture depended on twin type**



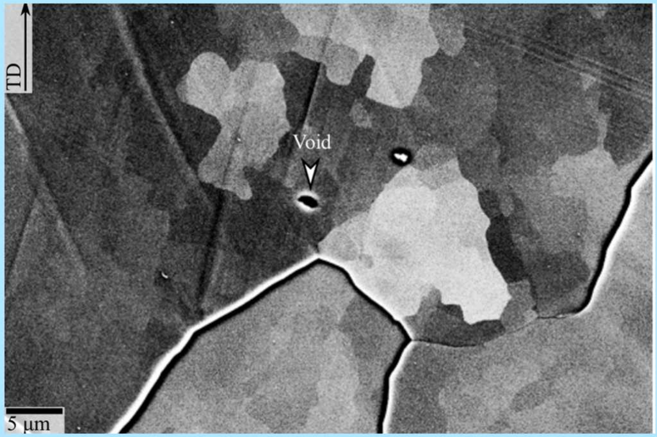
Warren, M. *et al., J. Nuc. Mat.,* 1968

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

SE Image: only slip bands visible



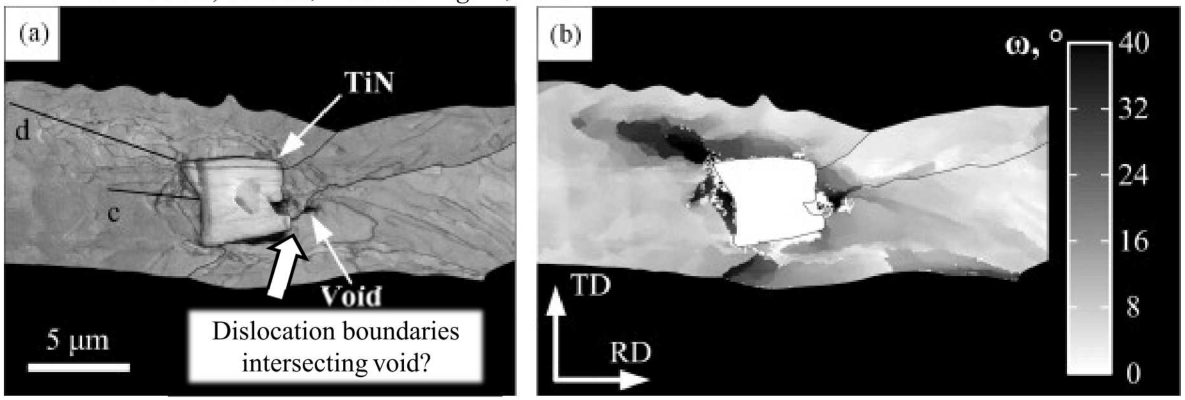
BSE Image needed to observe dislocation boundaries



- 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

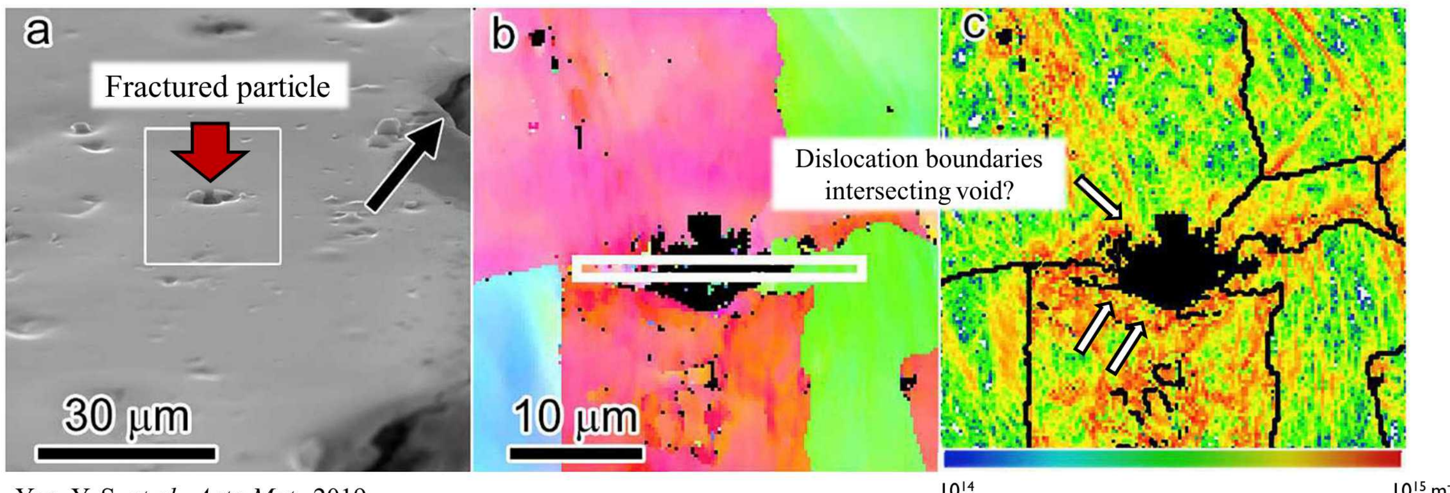
Relating particle-stimulated void nucleation to deformation-induced defect structures: dislocation boundaries

Leon-Garcia, O. *et al., Mat. Sci. Eng. A.*, 2010



Particle debonding at a TiN precipitate in an IF steel; EBSD data suggest that debonding was associated with a dislocation boundary, but data resolution is poor

Fracture of an intermetallic particle in Al6061, EBSD data suggest high density of dislocation boundaries intersected fractured particle



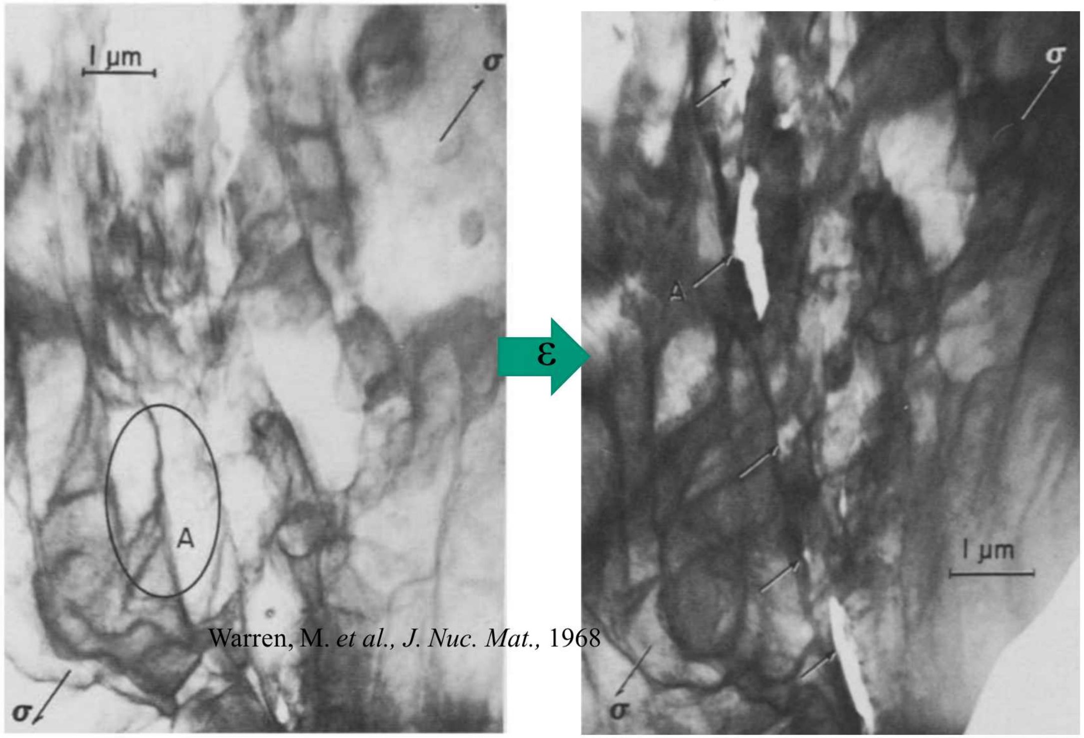
Yoo, Y. S. *et al., Acta Mat.*, 2019

Two prior studies contain examples of particle debonding and fracture near high-angle dislocation boundaries, but *no detailed characterization of the relationship between incipient voids and deformation defects was performed*

The microstructural features associated with particle-free void nucleation in single crystals

(a) The dislocation structure in a strained beryllium (HCP) single crystal

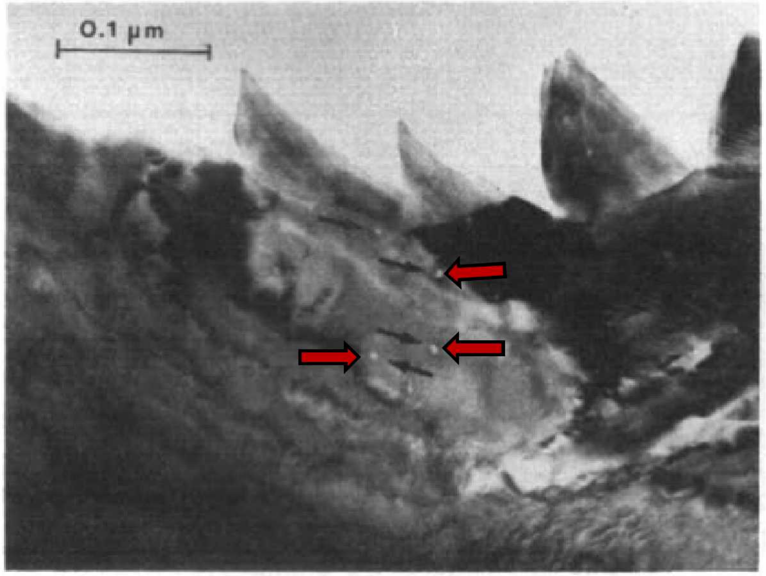
(b) The same region as in (a) after further straining. Voids along the dislocation boundary indicated with black arrows



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

Gardner, R.N., *et al.* "Crack initiation at dislocation cell boundaries in the ductile fracture of metals." *Materials Science and Engineering* 29.2 (1977): 169-174.

Vacancy clusters near the fracture surface of a single-crystalline, gold thin film

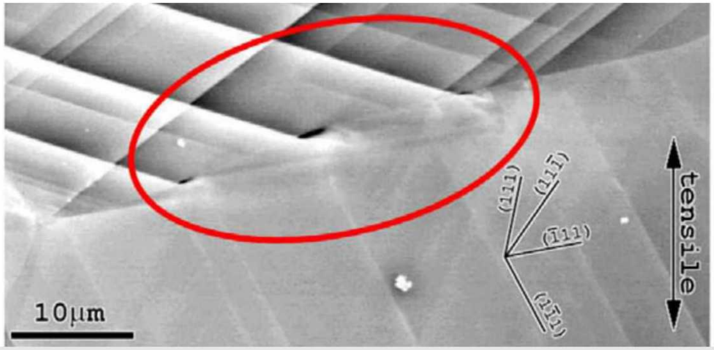


Wilsdorf, HGF. Acta Metallurgica, 1982

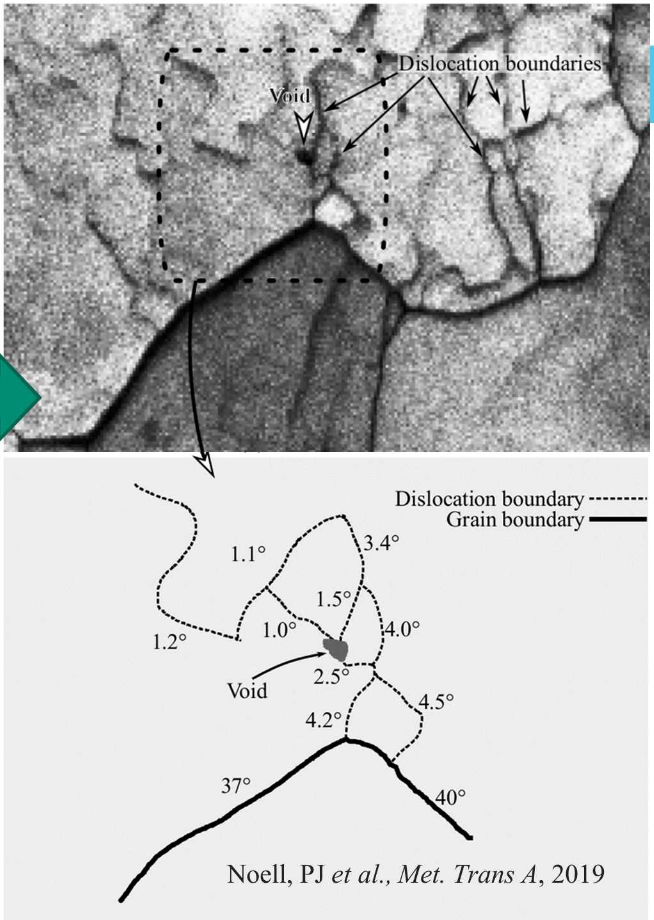
Early studies of the ductile rupture of single crystals demonstrated that voids nucleate at deformation-induced boundaries. Vacancy clusters observed near fracture surface in some materials. **Do dislocation boundaries and, maybe even vacancy clusters, play a role in void nucleation in bulk metals?**

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

Microcracks in TiAl correlated with twin intersections.

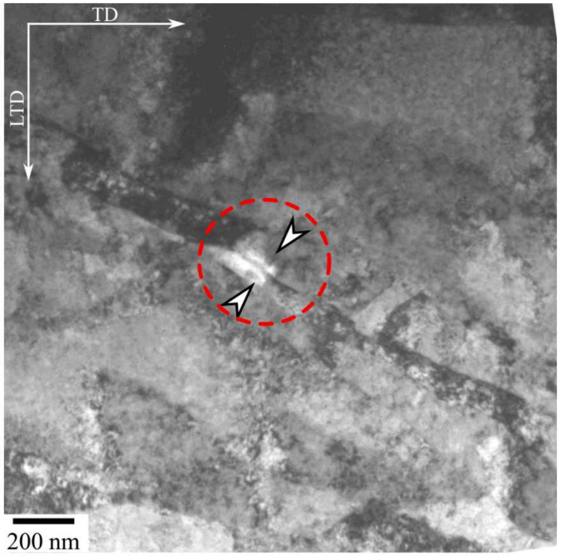


Void nucleation in Al occurred at dislocation cell walls, suppressed by DRX

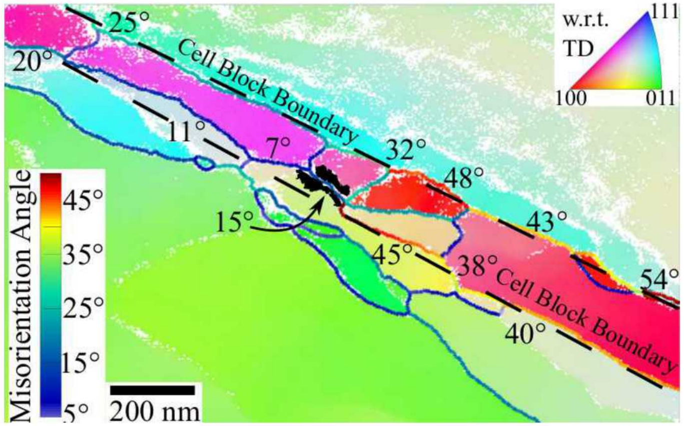


Noell, PJ *et al., Met. Trans A*, 2019

- In pure metals that deform by twinning, voids nucleate at grain-boundary/twin intersections and twin/twin intersections
- In both pure BCC (Ta) and FCC (Al) materials that deform by slip, voids nucleated at dislocation boundaries
- In high-purity Al, void nucleation was suppressed by dynamic recrystallization, which erased dislocation boundaries



Void nucleation in Ta primarily occurred at cell block boundaries

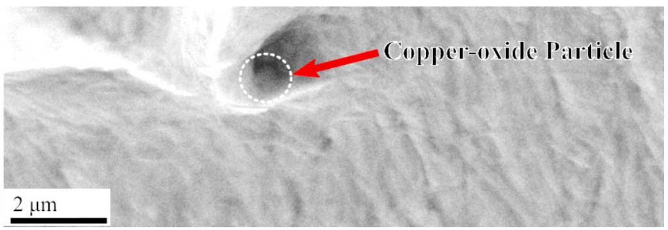


Noell, PJ et al., *Acta Mat,* 2017

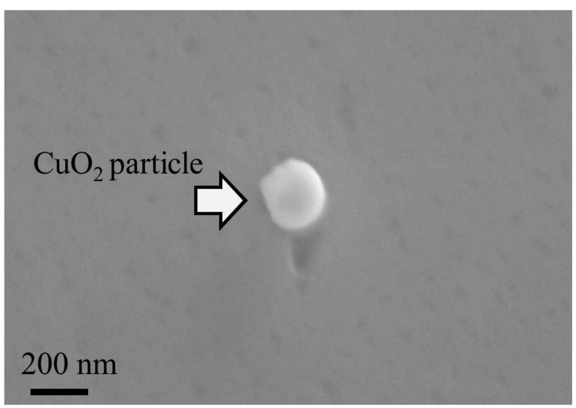
Do deformation-induced defect structures also enable the formation of microscale voids in materials that contain second-phase particles?

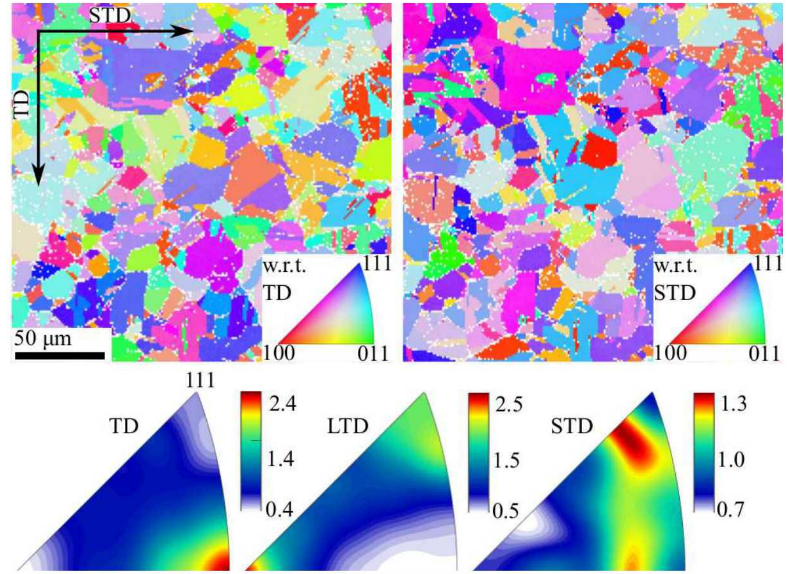
Material: 99.9% Cu sheet material, annealed after rolling

Contains ≈200 to 500 nm copper oxide inclusions, ≈1 inclusion per 8000 μm^3 of material



Images of (above) a CuO_2 particle on the fracture surface and (below) a CuO_2 particle within a void





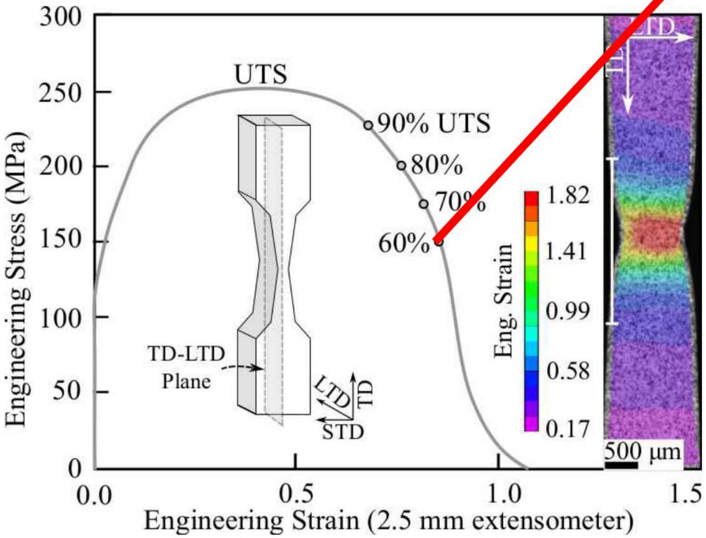
EBSD indicates that the as-received microstructure contained weakly-textured, equiaxed grains (8.7 μm diameter)

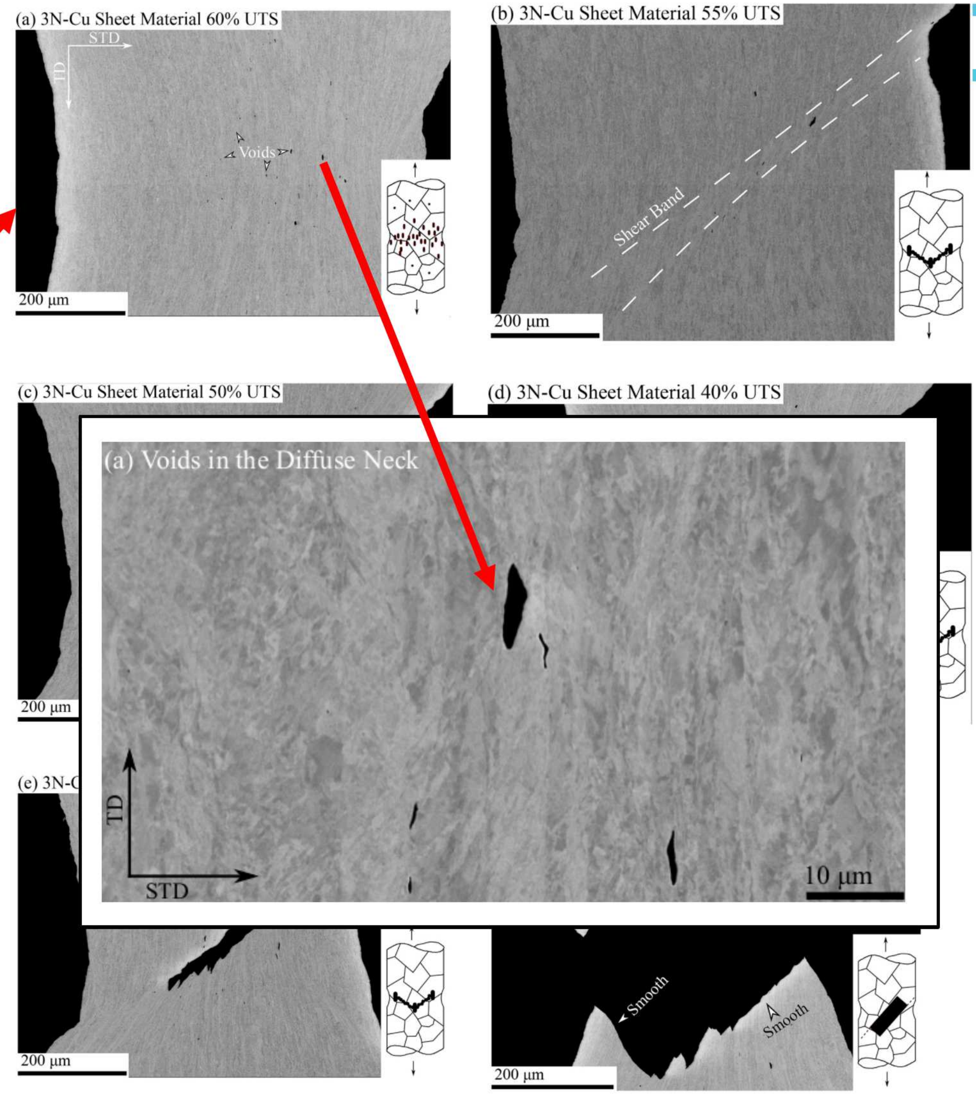
1. What deformation-induced defect structures are associated with the formation of microscale voids?
2. Do dislocation boundaries influence the formation of voids at inclusions?
3. Are copper oxide inclusions the primary nucleation site for voids in this material?

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

Ductile rupture of Copper

Noell, *et al., Acta Materialia* (2018)

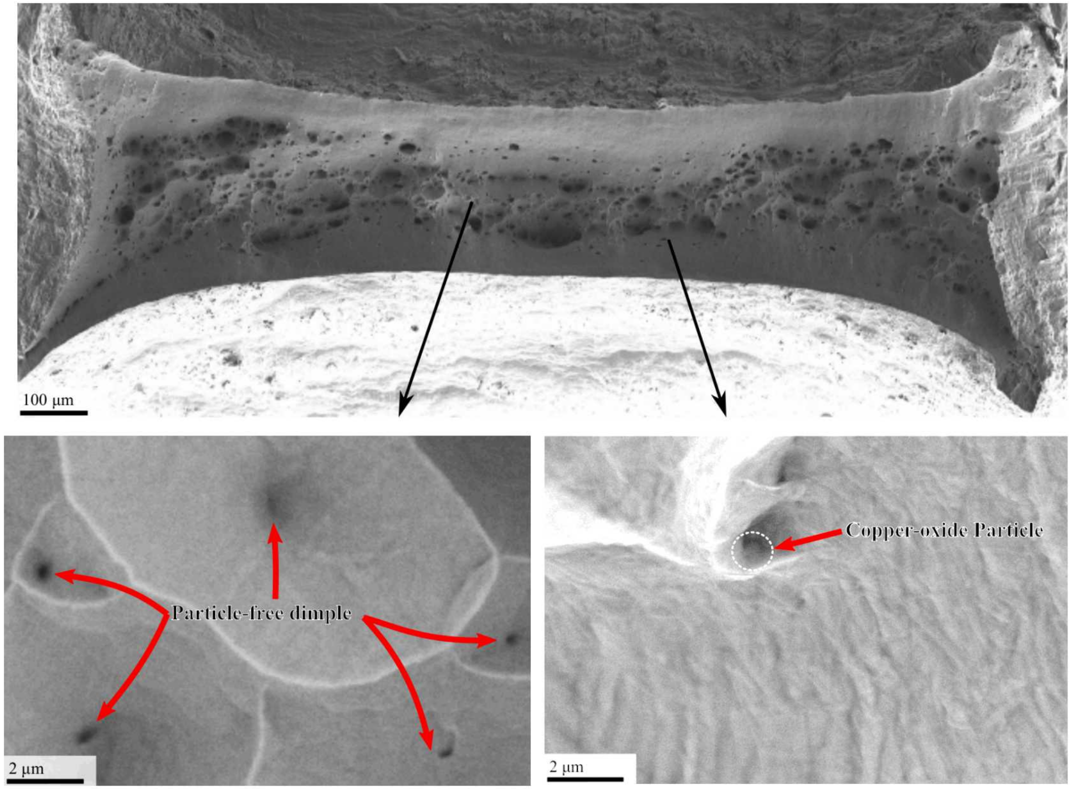




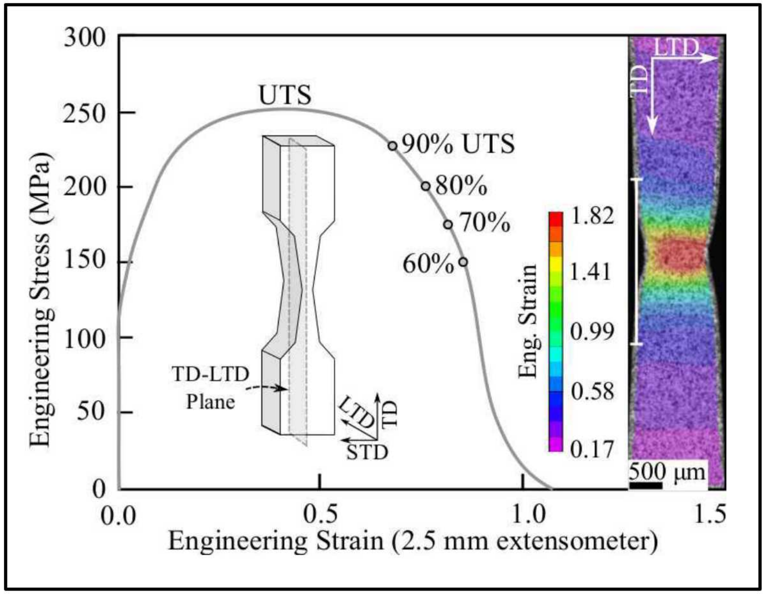
- Previous study using this material revealed that voids nucleate and grow within the diffuse neck of this material, coalesce along a shear band, and create an “Orowan void” that grows until failure

- ***But where do the “failure-relevant”, microscale voids come from?***

Producing incipient voids



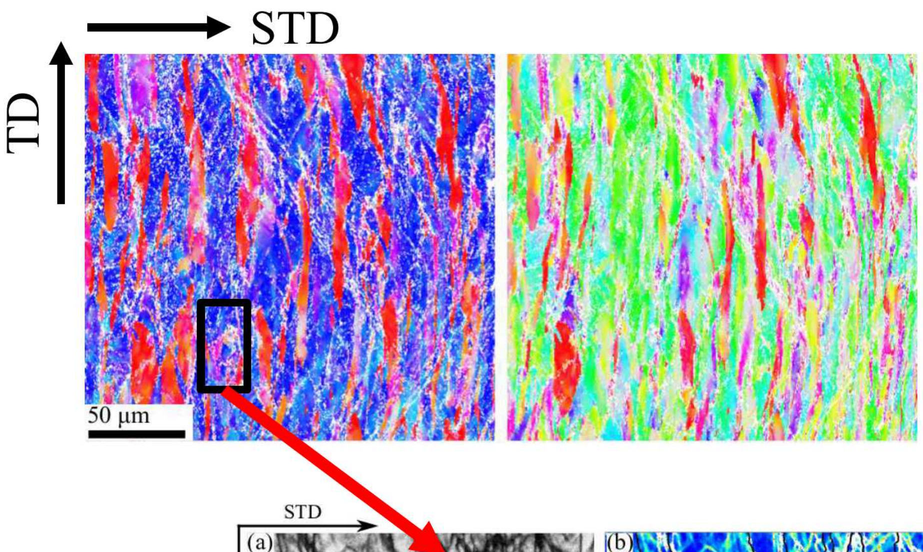
Inspection of the fracture surface suggests that most dimples were not associated with copper-oxide inclusions

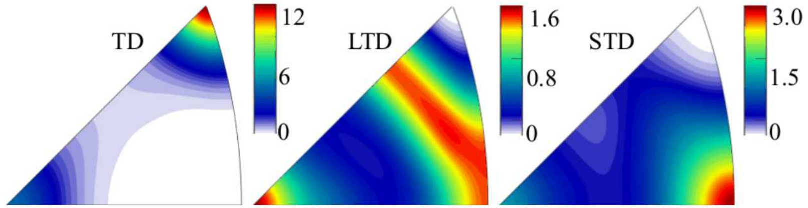


To understand the origins of these voids, interrupted tensile tests used to produce specimens containing incipient voids

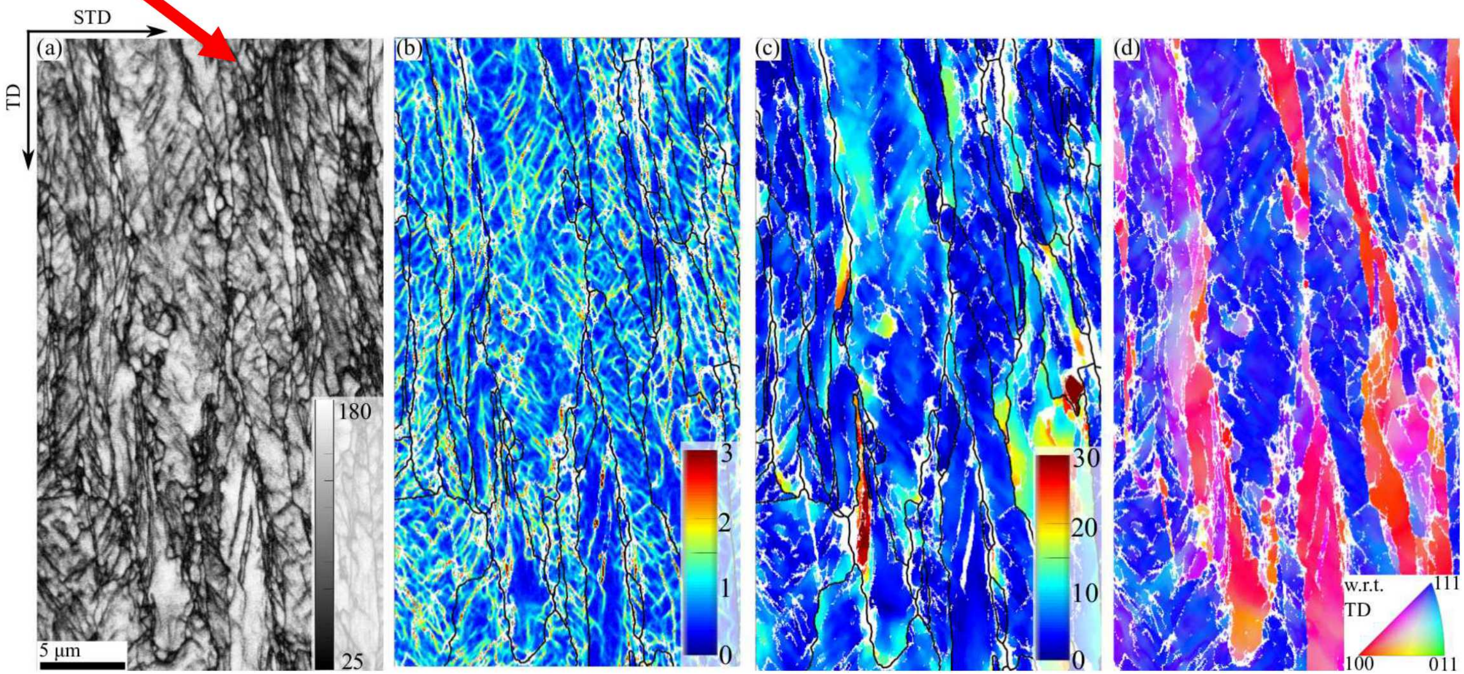
- Failure process
- **Defect structures in the deformed material**
- The origins of microscale voids

The deformed material (60% UTS): deformation boundaries



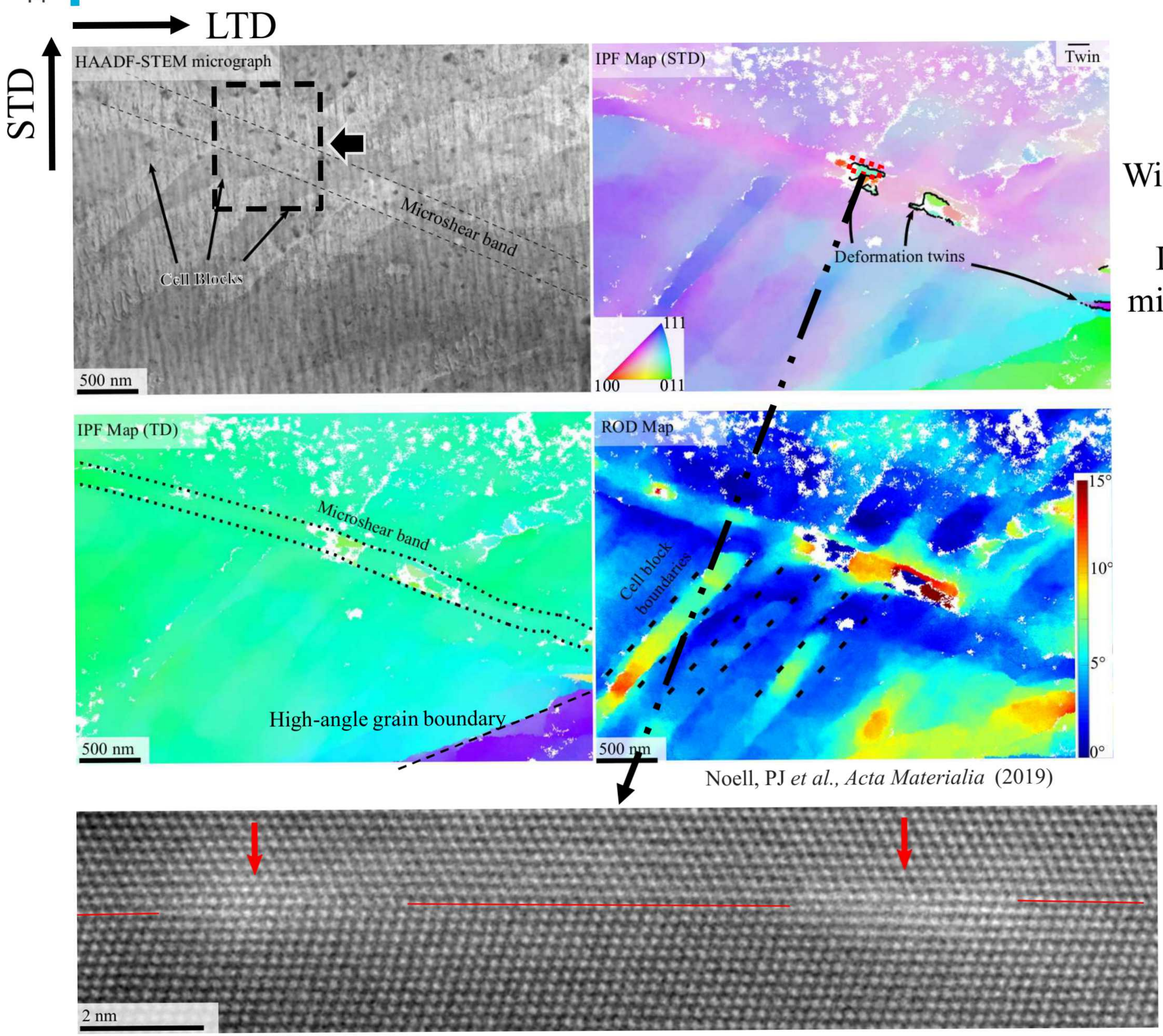


Deformation to 60% of the UTS created elongated grains (along TD), aspect ratio ≈4.4:1. Approximate strain in center of neck ≈150%-180%



EBSD data collected with a 50 nm stepsize reveal dislocation boundaries typical of heavily deformed FCC, including both elongated, lamellar boundaries and equiaxed cell structures

Deformation-induced defect structures at the microscale

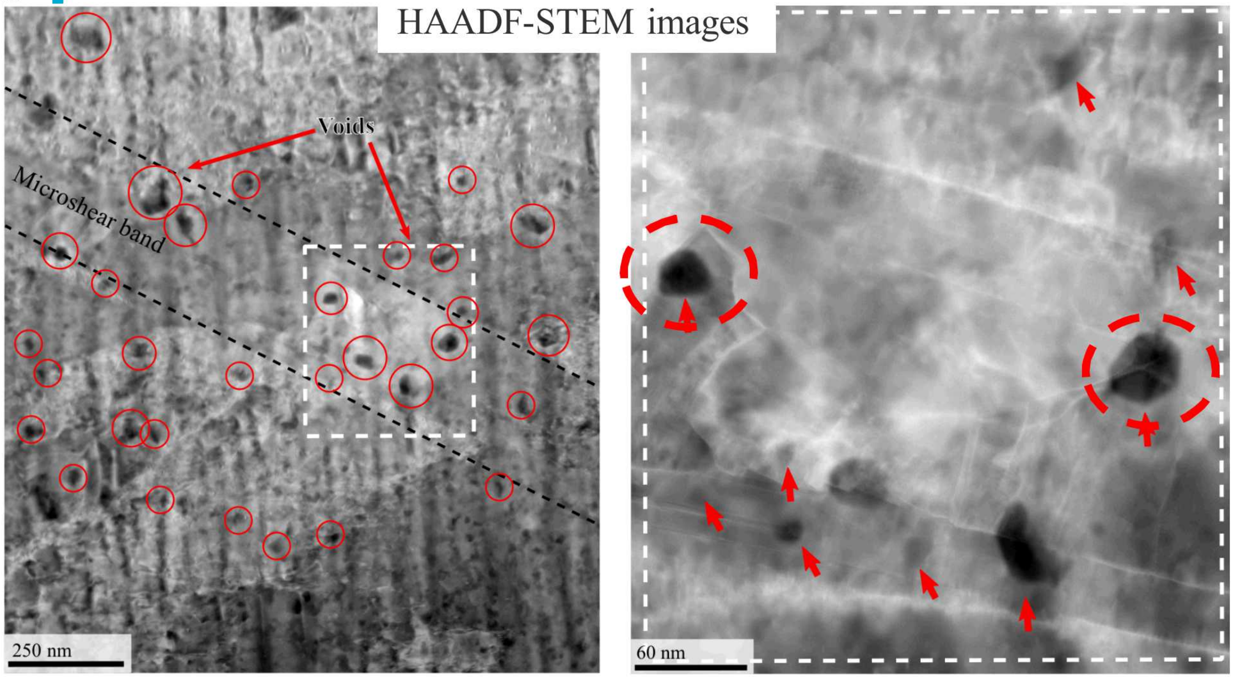


Noell, PJ *et al., Acta Materialia* (2019)

Within the diffuse neck of a 60% UTS specimen, Deformation twins and microshear bands observed at the microscale

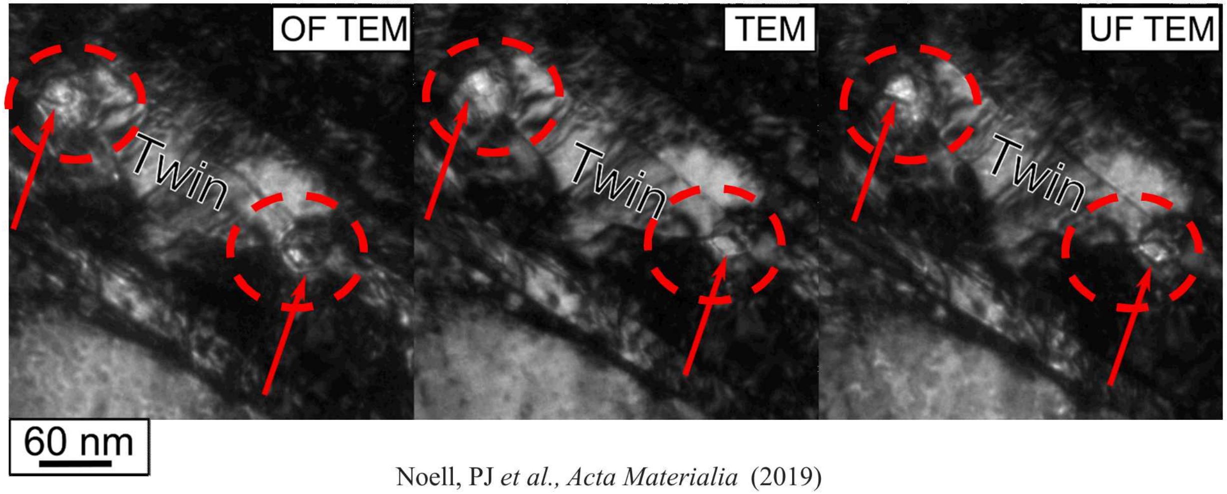
Atomic-resolution detail from HAADF-STEM image of twin boundary shows (a/6)<112> dislocations typical of deformation twins

Vacancy clusters/nanoscale voids



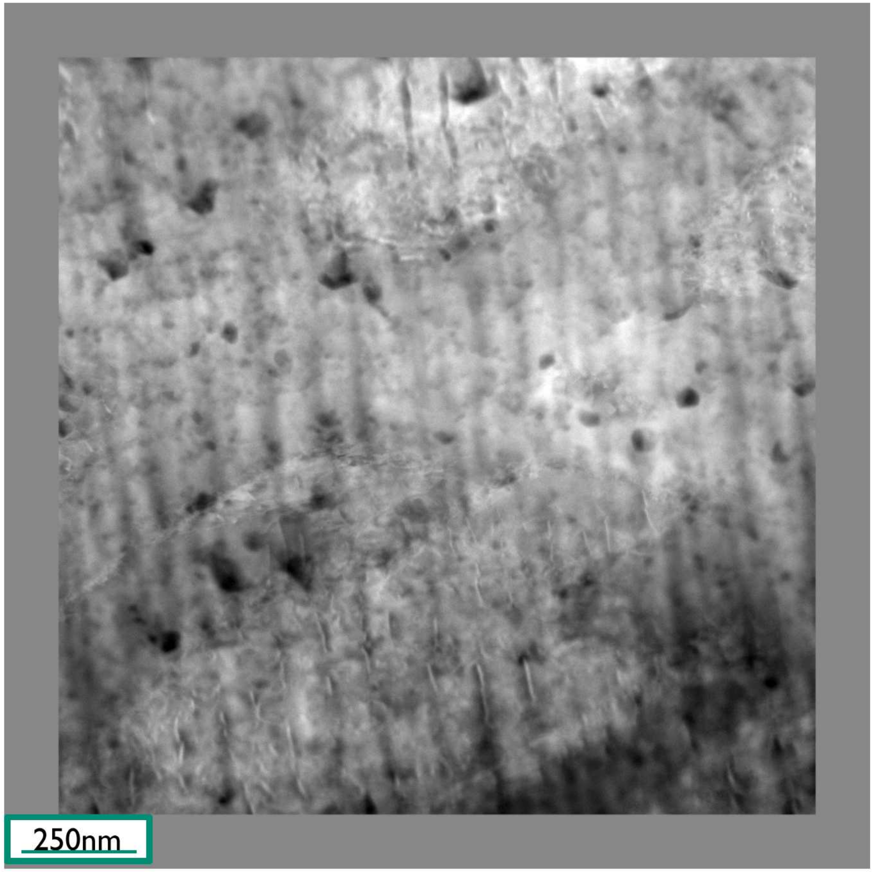
- Dozens of nanoscale (≈10-100 nm) voids observed both within and outside the microshear band
- Voids are faceted, similar to those created by vacancy condensation following quenching in other materials
- ***No evidence in EDS or diffraction measurements that suggests a change in composition or secondary-phase***

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

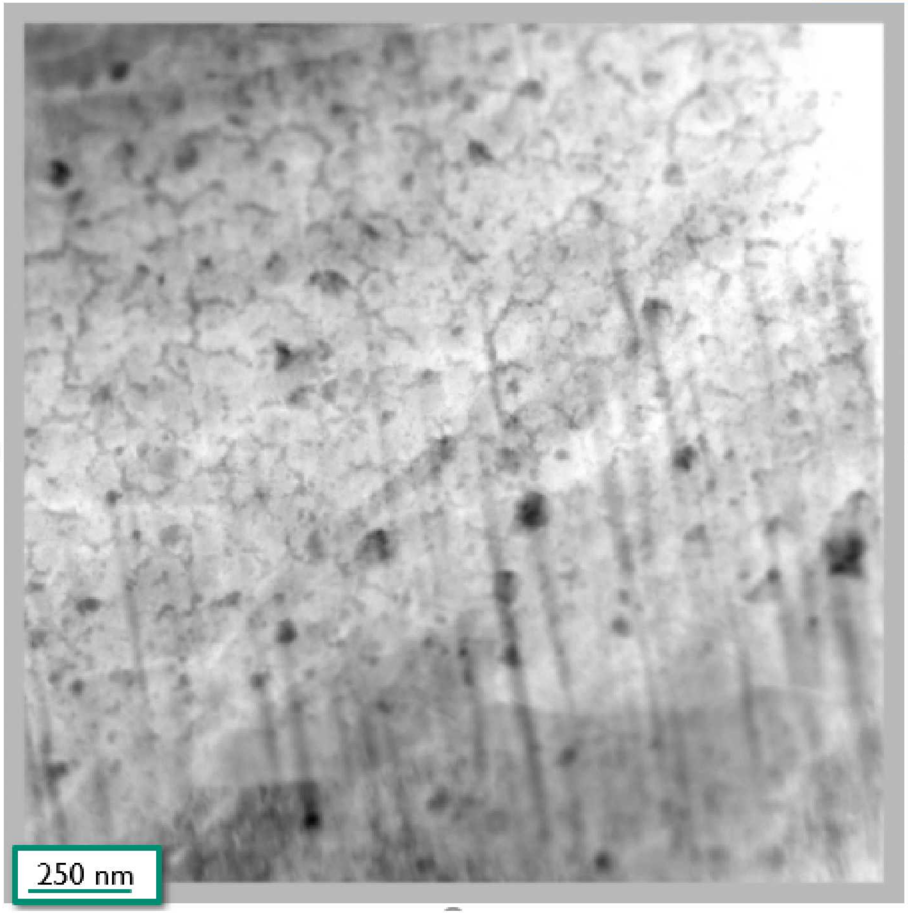


Noell, PJ *et al.*, *Acta Materialia* (2019)

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



A HAADF-STEM tomographic tilt series reveals mass-thickness contrast (indicative of voids) throughout the sample thickness.



Mass-thickness contrast, indicative of voids, also seen in HAADF-STEM of a second TEM foil (also extracted from the midplane, necked gauge region of a 60% UTS sample)

- Estimate from these micrographs suggests a density of 10^2 per um^2 nanoscale voids
- Observed both within microshear band and outside, suggesting they may be 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

← (Particle decohesion, Particle delamination, Grain boundary cleavage) Void nuclei will be distributed heterogeneously at specific microstructural features

← (Vacancy condensation) Void nuclei will be fairly homogeneous

- Experimental measurements of Cu at $\varepsilon = 60\%$ 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 $\approx 10^{-4}$

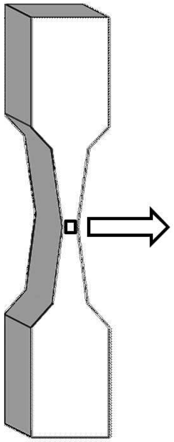
$$c_v = \frac{A}{G}\int_0^{\varepsilon} \sigma \, d\varepsilon$$

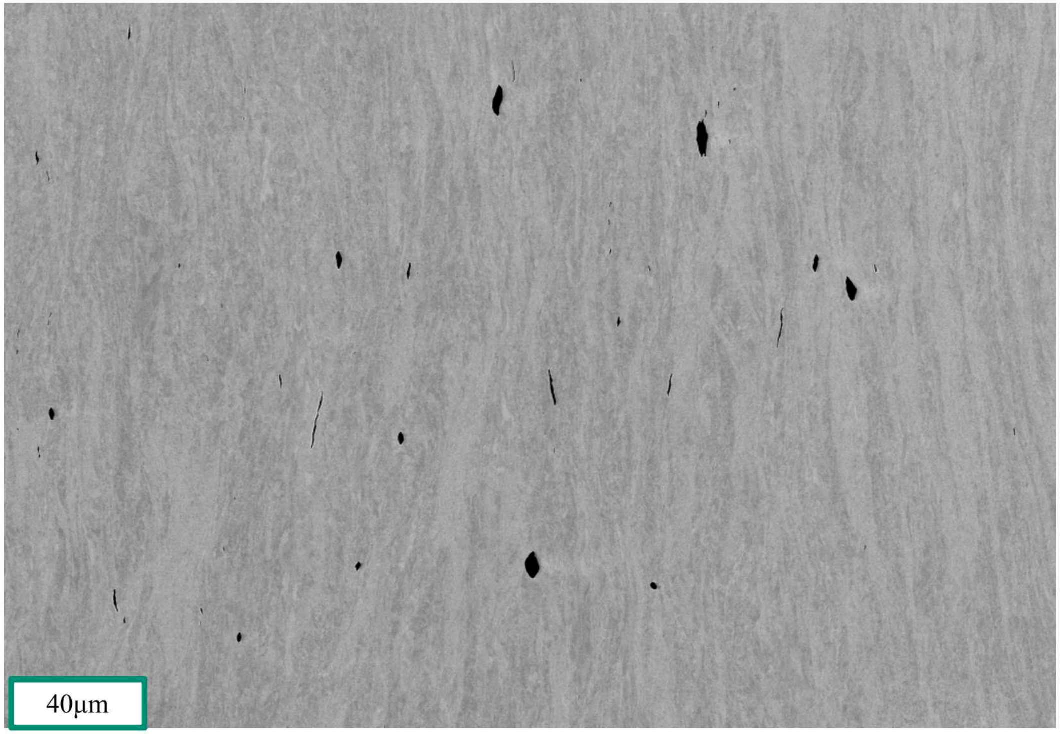
C_v - vacancy concentration
A - constant (A~0.1)
G - shear modulus

- 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

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

What happens to the nanoscale voids?





Area density of microscale voids in the center of the neck of a 60% UTS sample was ≈ 1 per 400 µm^2. Conservatively, only 1 in 10,000 nanoscale voids grows to the microscale. ***Void nuclei thus appear to be overseeded!***

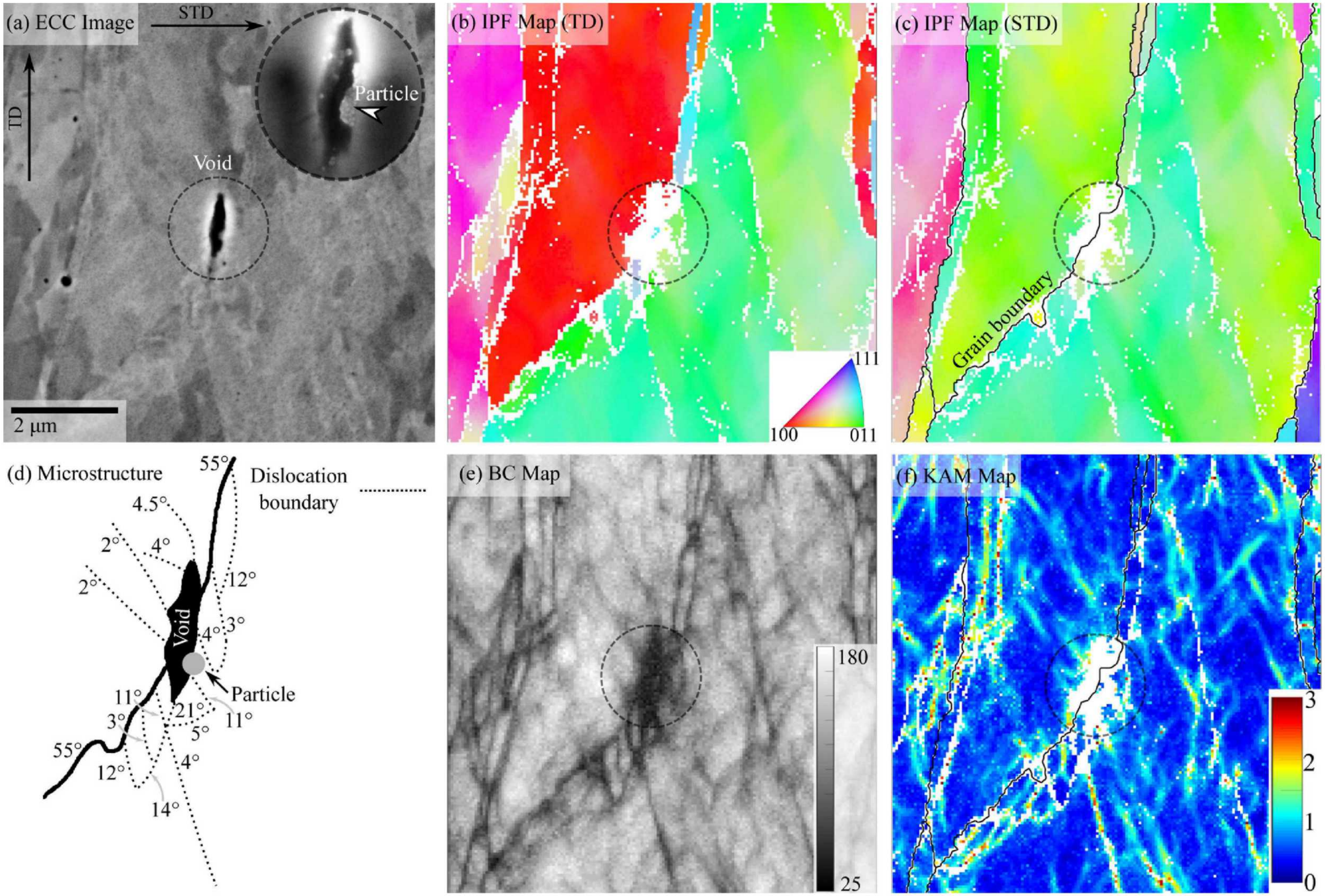
Why do some voids grow while the vast majority stagnate?

The microstructural features associated with microscale voids

22 incipient (< 2 µm) voids characterized using a combination of EBSD (20) and TEM (2)

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

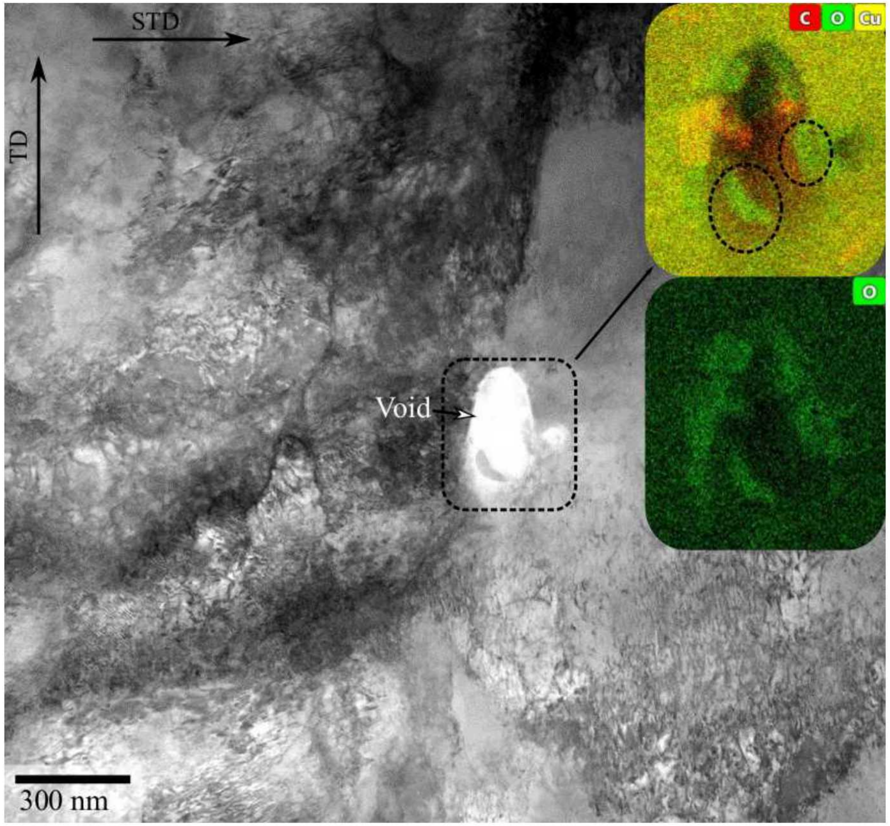
Particle-stimulated voids: microscale

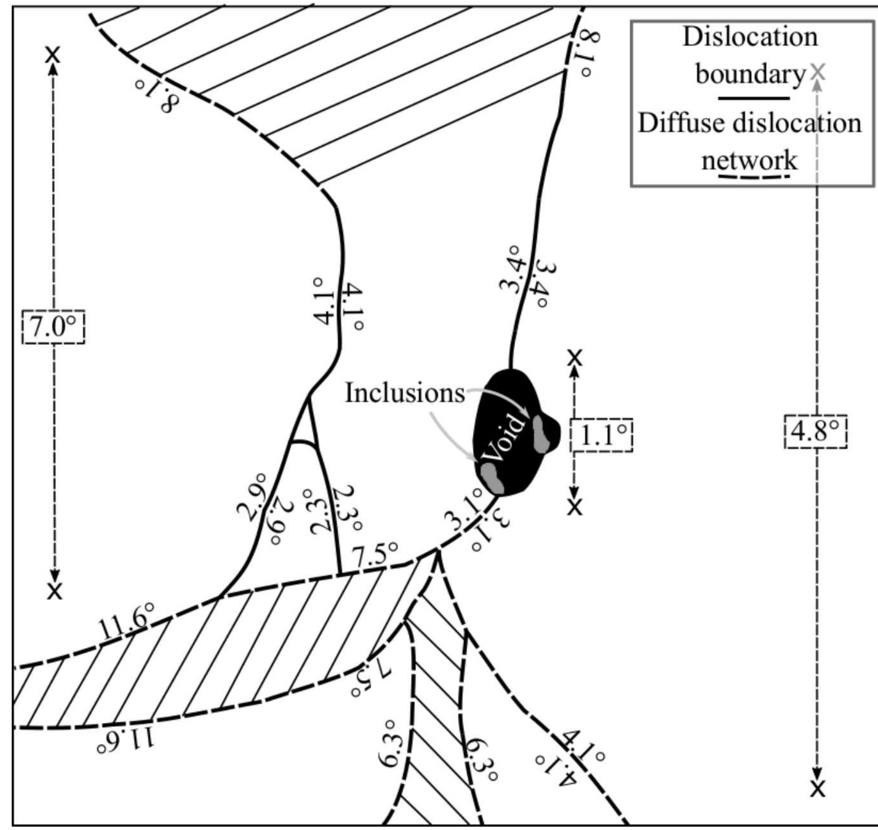


Typical void that nucleated at a particle is shown

- Intersected by multiple cell block boundaries

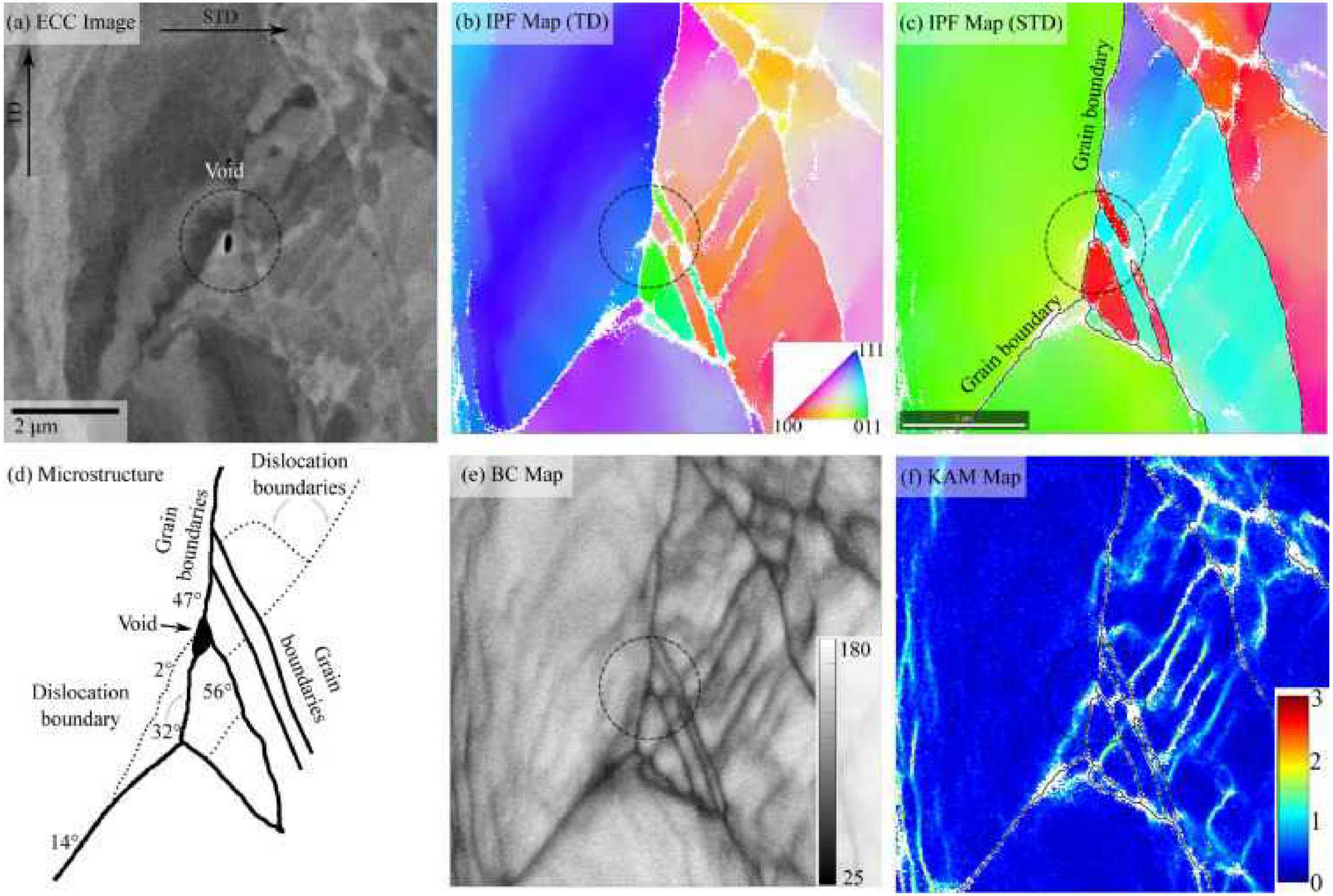
Particle-stimulated voids: nanoscale





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

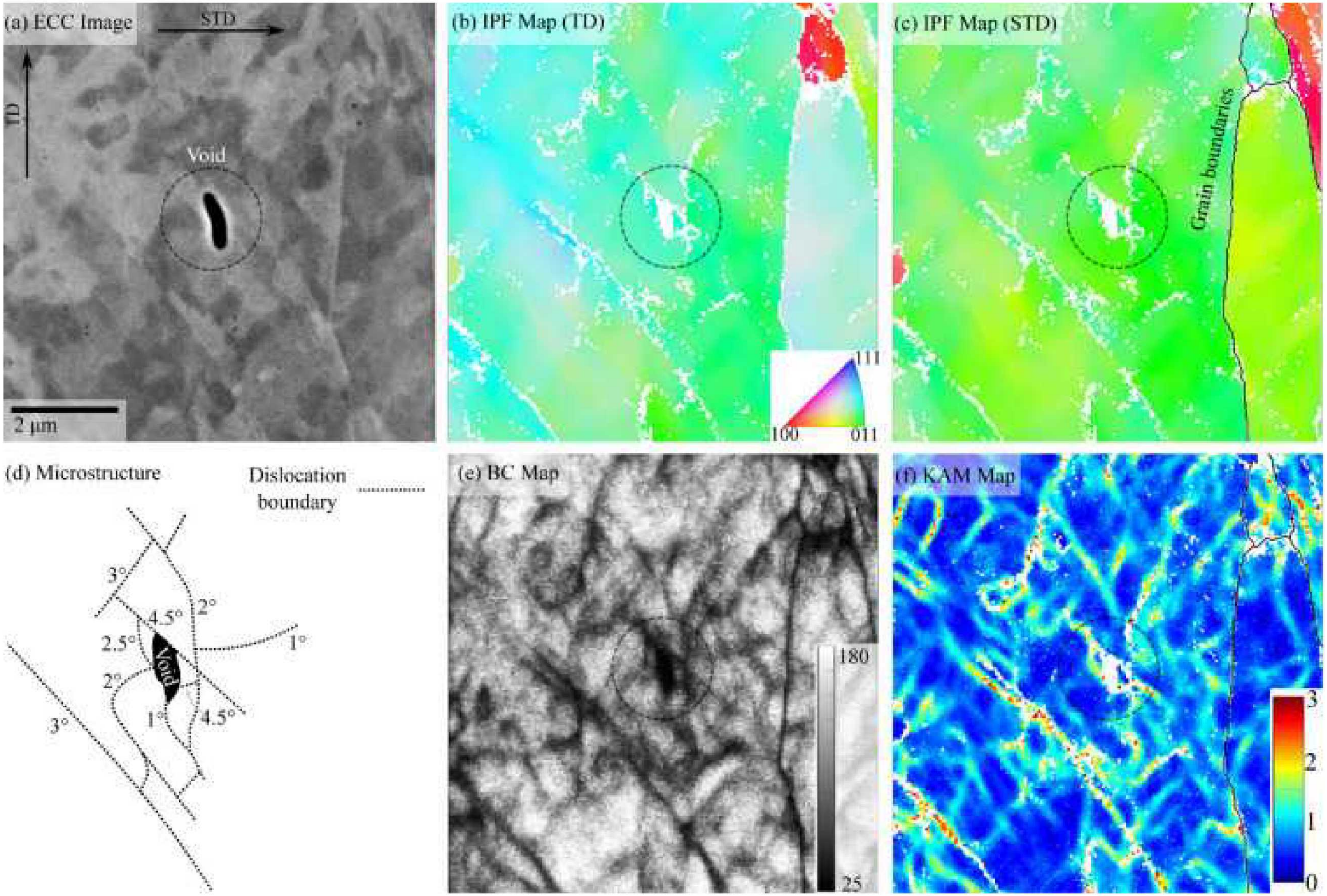
Grain-boundary, inclusion-free voids



Typical void that nucleated at a grain boundary is shown

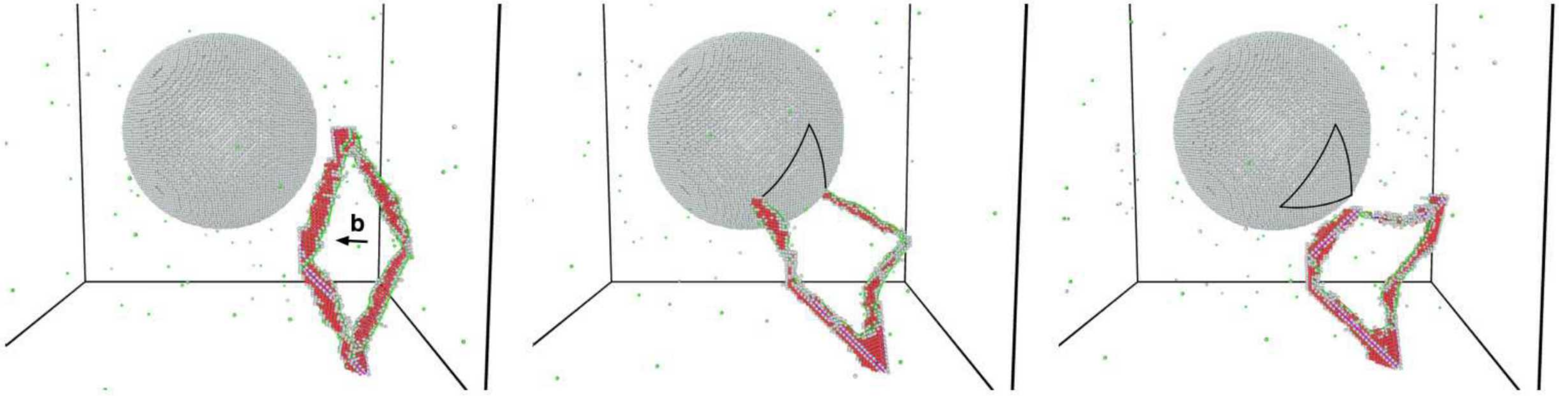
- Void emerged at the intersection between a grain boundary triple point and a cell block boundary

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



(a) (b) Sills, R. *et al., MRL* (2019) (c)

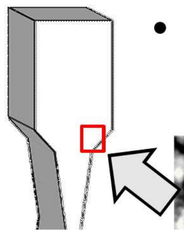
- MD simulations of void growth by dislocation absorption suggest that this is a realistic mechanism for the growth of nanoscale voids
- This mechanism is only realistic if the nanovoid is surrounded by a high-density of mobile dislocations, which is expected if the nanovoid is intersected by one or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

Conclusions

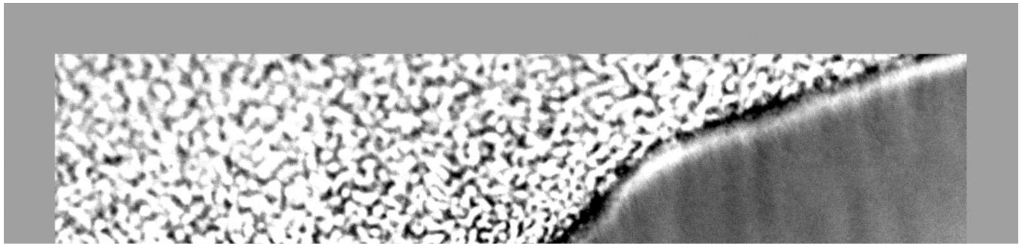
1. At what microstructural features do microscale voids originate? ***Are inclusions the primary nucleation site?***
2. What deformation-induced defect structures are associated with the formation of microscale voids at inclusions?

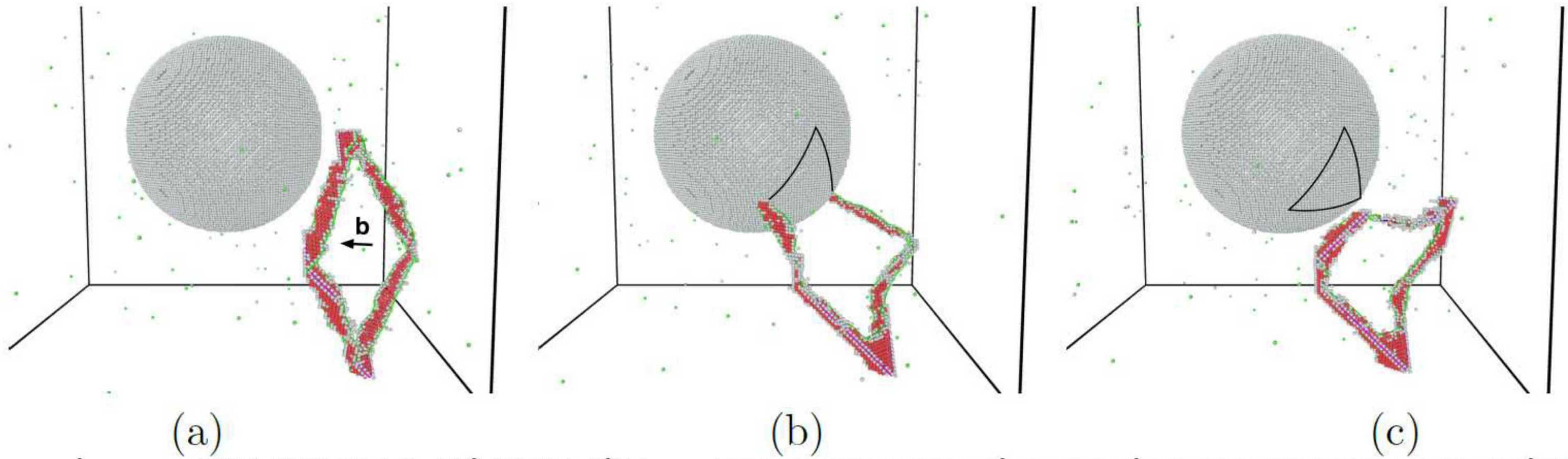
- Voids nucleated at the nanoscale, perhaps by vacancy condensation, and were distributed fairly homogeneously within the midplane of the necked gauge region
- ***Void nuclei appear to be overseeded.*** The formation of microscale voids thus controlled by the emergence of nanoscale voids to the microscale (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.***

Are “vacancy clusters” just a specimen preparation artifact?



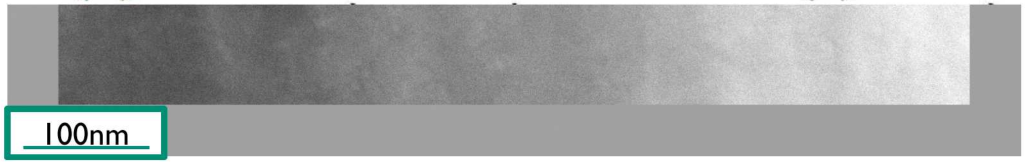
- TEM foils extracted from fillet between gauge and grip





away from the Ga+ rich surface layer.

This observation suggests that any nanoscale defect content smaller than ≈3 nm may be an artifact of specimen preparation, but that ***the >10 nm voids observed in the deformed microstructure are real, intrinsic features of the microstructure***



Extremely little mass-thickness contrast observed in tomographic series. Some surface damage is visible at the extreme tilts (central feature due to electron beam depositing material).