# [1][WPA Name as listed in SSPS]

Mechanisms for Ductile Rupture

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## Abstract and Introduction

Ductile rupture in metals is generally a multi-step process of void nucleation, growth, and coalescence. Particle decohesion and particle fracture are generally invoked as the primary microstructural mechanisms for room-temperature void nucleation. However, because high-purity materials also fail by void nucleation and coalescence [1, 15, 22], other microstructural features must also act as sites for void nucleation. Early studies of void initiation in high-purity materials, which included post-mortem fracture surface characterization using scanning electron microscopy (SEM) and high-voltage electron microscopy (HVEM) and in-situ HVEM observations of fracture, established the presence of dislocation cell walls as void initiation sites in high-purity materials [22]. Direct experimental evidence for this contention was obtained during in-situ HVEM tensile tests of Be single crystals. Voids between 0.2 and 1 μm long appeared suddenly along dislocation cell walls during tensile straining [10, 19]. However, subsequent attempts to replicate these results in other materials, particularly α -Fe single crystals, were unsuccessful because of the small size of the dislocation cells [12], and these remain the only published in-situ HVEM observations of void nucleation at dislocation cell walls in the absence of a growing macrocrack. Despite this challenge, other approaches to studying void nucleation in high-purity metals also indicate that dislocation cell walls are nucleation sites for voids. In-situ HVEM studies of crack growth in Ag single crystals found that voids initiated at regular intervals in the most heavily work-hardened area of the crystal ahead of the advancing crack [2, 18, 22]. Post-mortem HVEM studies of crack flanks of Au polycrystals tensile specimens revealed microvoids, stacking fault tetrahedra, and dislocation cell walls in the crack flank [21]. The presence of these features at all examined crack flanks lead Wilsdorf to conclude that these were responsible for void nucleation [21]. Similarly, dislocation cells with misorientations as large as 35 across the walls were observed in crack flanks of α -Fe single crystals [12]. Gardner et al. calculated that these cell walls were an energetically preferred site for void initiation in α -Fe single crystals, though this hypothesis could not be directly confirmed because of the small scale of the dislocation cells [11, 12]. Together, these data suggest that work-hardening before fracture

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creates dislocation cells whose boundaries act as nucleation sites for voids in pure materials, regardless of crystal structure.

Wilsdorf and coworkers demonstrated that dislocation cell walls can act as sites for void initiation in materials containing second-phase particles [22]. Measurements of dimple and second-phase particle spacing on the fracture surfaces of 304 SS and Al-1.79 wt.% Cu specimens revealed that the ratio of dimple spacing to particle spacing was considerably larger than unity [2, 1, 8, 7]. In-situ HVEM tensile tests of 304 SS and Al-Cu alloys containing either θ or θ ' precipitates revealed that voids nucleate ahead of the growing crack at regularly spaced intervals. This regular spacing makes it unlikely that second phase particles are exclusively responsible for void nucleation in these materials [2, 7, 22]. HVEM observations confirmed that θ and θ ' precipitates are not generally associated with void initiation sites in the Al-Cu alloys [8, 7]. Energy dispersive X-ray analysis of dimples on the fracture surface of bulk 304 SS tensile specimens also indicated that second-phase particles were not generally present in the void [1, 2].

Because most early studies of void nucleation in pure metals by Wilsdorf and coworkers used single crystals, though, it is not clear their observations of void nucleation at dislocation cell walls are applicable to polycrystalline materials. Intriguingly, although voids initiated at dislocation cell walls in Be single crystals, voids instead initiate at grain boundaries in polycrystalline Be [20]. Submicron-sized voids were also observed at grain boundaries in commercial-purity Ti and near-gamma TiAl samples that were strained in 4-point bending to 2 and 1.4%, respectively [16, 5, 4]. Alternatively, recent post-mortem EBSD data from Ta tensile specimens elongated to 60% of their ultimate tensile strength revealed the presence of regularly spaced voids at or near subgrain boundaries [6].

Because moving dislocations produce vacancies [14, 17, 2], vacancy clusters at dislocation cell walls could participate in void nucleation. The discovery of microvoids in crack flanks of Au [21] support a vacancy coalescence mechanism. Cuttino et al. [9] also demonstrated that void nucleation by vacancy condensation is energetically possible in single crystals of FCC metals. It remains to be conclusively demonstrated, though, that vacancy condensation plays a primary role in void nucleation at dislocation cell walls [22]. Alternatively, the strain energy created by misorientations between dislocation cells in single crystals of α-Fe and Be is sufficiently large to favor void nucleation at dislocation cell walls [10, 12]. Similarly, the projected energy density of dislocation cell walls in Al-Cu alloys containing second-phase particles was approximately an order of magnitude larger than that associated with the second-phase particles [8]. These results suggest that void initiation at dislocation cell walls is primarily the result of the strain energy rather than the vacancies associated with the cell wall [13], though vacancy clusters may provide a secondary mechanism for void initiation or decrease the energy necessary for void nucleation at a dislocation cell wall [22]. According to this theory, void initiation at a dislocation cell wall depends primarily on the strain energy associated with that wall.

**Approach**

The current study examined ductile rupture in 99.9% tantalum. No voids or inclusions were found in examinations of this as-received material by either SEM or TEM observation. Single crystal tantalum

tensile specimens with [111], [100], and [110] crystal orientations relative to the loading axis were prepared by electro discharge machining (EDM). The sample geometries were similar to those used in previous work [5]. To summarize, the aspect ratio of the gage geometry is comparable to the ASTM E8 standard, but several times smaller. Mechanical testing was performed in displacement-control mode using a custom tensile testing apparatus. The oriented single crystal specimens were tested in situ inside of a SEM.

**Results and Impacts**

Tensile stress-strain curves for the different single-crystal orientations are shown in Figure 1. The differences in observed yield strength and work hardening behavior was generally consistent with prior studies on single crystal tantalum. However, those prior studies did not interrogate the damage state associated with failure. There are substantial differences in the damage state prior to catastrophic failure. Two key examples are taken from the [110] (Sample 8) and [111] (Sample 7) tensile loading directions. The [110] orientation exhibited delayed necking and geometric softening. However, SEM inspection of mid-plane cross-sections (upper right in Figure 1) showed extensive void coalescence damage. By contrast, the [111] orientation showed early necking and extensive geometric softening associated with the development of a hourglass-shaped neck. A complementary midplane cross-section (lower right in Figure 1) shows the presence of the deep neck (black feature at bottom), but no signs of damage nucleation. The lack of pronounced internal damage is especially surprising since the [111] condition had undergone such extensive geometric softening, associated with ~90% loss in load-carrying capacity.

To understand these two very different damage states, electron backscattered diffraction (EBSD) was performed on the midplanes, corresponding to the images in Figure 1. The EBSD crystallographic orientation maps corresponding to the two samples highlighted in Figure 1 is shown in Figure 2. There is a remarkable feature that is present for the [110] condition (left panel of Figure 2): a sharp high-angle grain boundary with subgrains that were oriented with [100] relative to the tensile axis (red pixels). This sharp high angle grain boundary was notably absent in the [111] condition (right panel).

Interestingly, Sample 7 with an initial [111] crystal orientation underwent a large crystal reorientation in the vicinity of the neck to reorient to a [110] orientation relative to the tensile axis. It is unclear why the deformation-induced [110] orientation does not proceed to form [100] subgrain boundaries and fail in a manner similar to Sample 8.

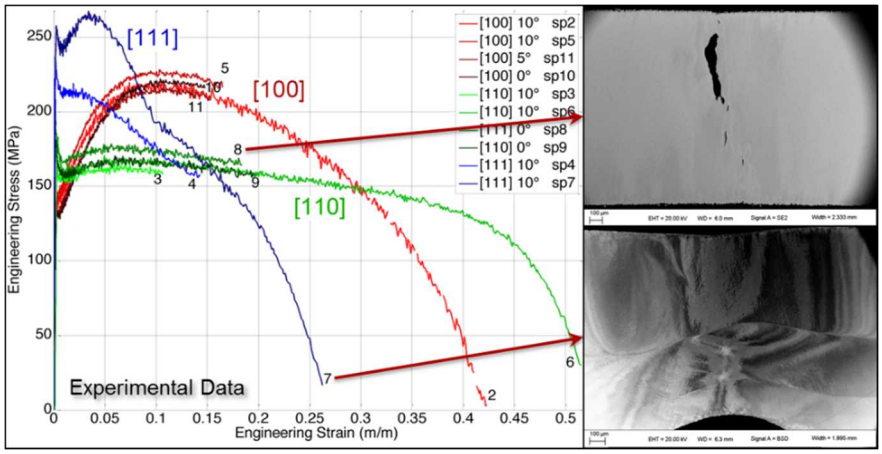


*Figure 1. Stress-strain behavior of different single-crystal orientations along with corresponding damage morphology.*

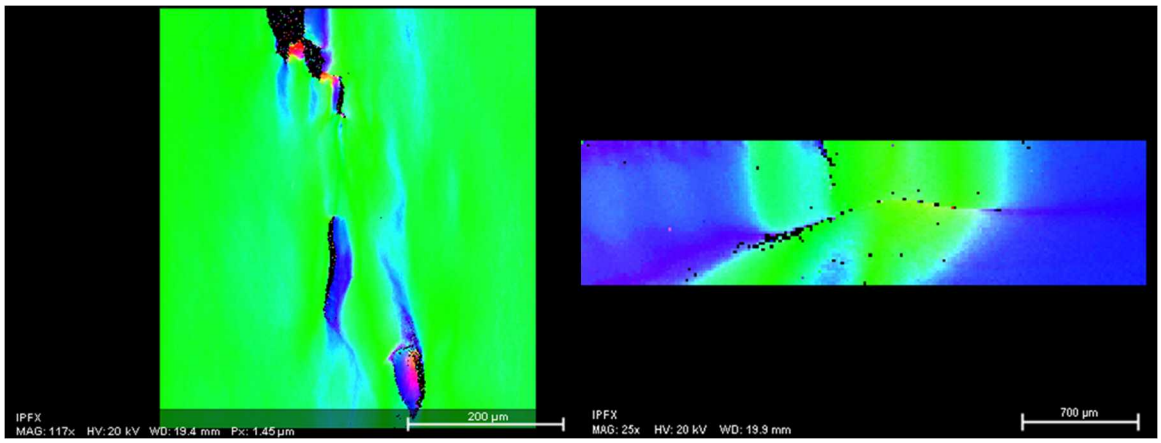


*Figure 2. (left) EBSD inverse pole figure crystallographic orientation maps for (left) Sample 8 [110] tensile orientation, and (right) Sample 7 [111] tensile orientation. In both cases, the tensile loading axis was horizontal relative to the frame of the picture. Crystal orientation was relative to the tensile axis (IPF-X).*

**Conclusions and Future Work**

The preliminary evidence presented in the current study further strengthens the hypothesis that the formation of some types of sharp high-angle subgrain boundaries is a precursor to void nucleation in pure tantalum. Future work will investigate the role that the dislocation cell walls at the deformation-induced high-angle boundary play in void nucleation. Finally, if additional evidence continues to support the hypothesis of high-angle boundary formation as a precursor to void nucleation, it will be important to develop a constitutive model for this phenomenon and validate its applicability in other cubic pure metals.

**Summary of Findings and Capabilities Related to Aging**

Pure single crystal tantalum under quasistatic tensile deformation exhibits very different damage progression depending on the crystal orientation. With an initial [110] orientation relative to the tensile axis, voids appeared at locations of sharp high-angle subgraph formation. With an initial [111] orientation, no voids were formed, but the crystal exhibited substantial rotation from [111] to [110]. These observations, taken in combination with other similar observations, will serve as the basis for the development of a mechanistically-informed constitutive model for ductile rupture in polycrystalline metals.

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