



## Full length article

## Do voids nucleate at grain boundaries during ductile rupture?



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## ARTICLE INFO

## Article history:

Received 11 May 2017

Received in revised form

29 June 2017

Accepted 1 July 2017

Available online 12 July 2017

## Keywords:

Ductile fracture

Cavity nucleation

Deformation structure

Fracture mechanisms

Void growth

## ABSTRACT

In the absence of pre-existing failure-critical defects, the fracture or tearing process in deformable metals loaded in tension begins with the nucleation of internal cavities or voids in regions of elevated triaxial stress. While ductile rupture processes initiate at inclusions or precipitates in many alloys, nucleation in pure metals is often assumed to be associated with grain boundaries or triple junctions. This study presents *ex situ* observations of incipient, subsurface void nucleation in pure tantalum during interrupted uniaxial tensile tests using electron channeling contrast (ECC) imaging, electron backscatter diffraction (EBSD), transmission Kikuchi diffraction (TKD) and transmission electron microscopy (TEM). Instead of forming at grain boundaries, voids initiated at and grew along dislocation cell and cell block boundaries created by plastic deformation. Most of the voids were associated with extended, lamellar deformation-induced boundaries that run along the traces of the {110} or {112} planes, though a few voids initiated at low-angle dislocation subgrain boundaries. In general, a high density of deformation-induced boundaries was observed near the voids. TEM and TKD demonstrate that voids initiate at and grow along cell block boundaries. Two mechanisms for void nucleation in pure metals, vacancy condensation and stored energy dissipation, are discussed in light of these results. The observations of the present investigation suggest that voids in pure materials nucleate by vacancy condensation and subsequently grow by consuming dislocations.

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## 1. Introduction

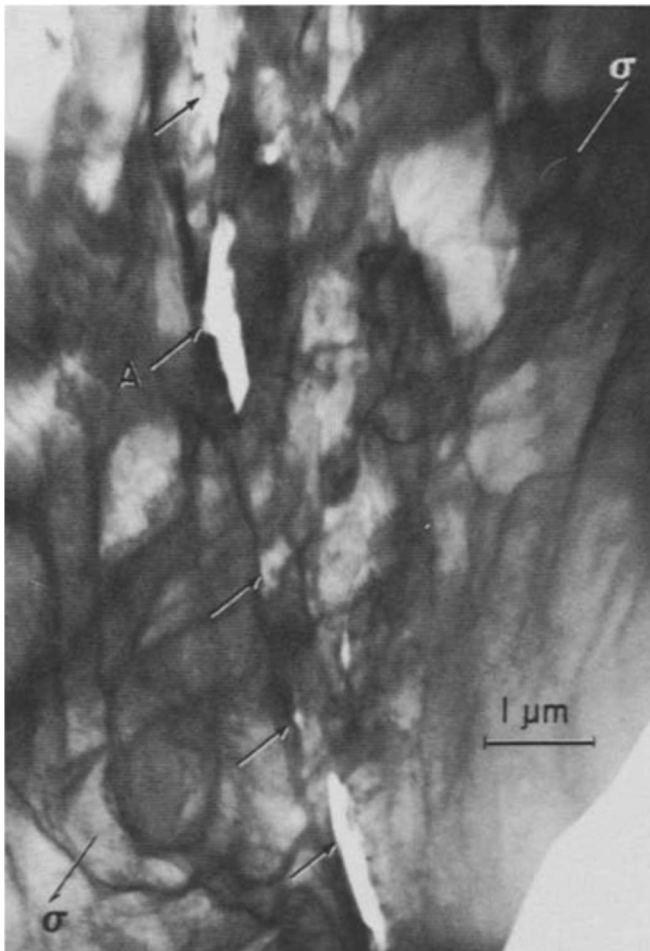
In the middle of the 20th century, the pioneering work of Tipper [1] and Puttick [2] revealed that ductile fracture is a multi-step process of void nucleation, growth and coalescence. Subsequent modeling and experimental work based on the theories of Rice [3], Gurson [4], Tvergaard [5,6] and Needleman [7,8] produced well-validated models for the growth and coalescence of voids during tensile plastic deformation under high-triaxiality conditions, *e.g.* Refs. [9–12]. There is, however, still no broad consensus among these models on the appropriate criteria for void nucleation. Many models assume that the metal contains a preexisting population of voids [10,13,14], but this is rarely observed experimentally in engineering materials. More sophisticated approaches invoke inter-phase/interface decohesion and/or particle cracking as the mechanisms of void nucleation [7,15,16]. Although these approaches are often useful, they are not always applicable to metals with submicron particles [11,17–22] and are clearly incorrect for

pure, single-phase metals. Because these materials also fail by void nucleation and coalescence [23–25], an essential first step to understanding their fracture is determining where and how voids nucleate in the absence of second-phase particles.

During quasistatic loading, voids in pure, low-stacking fault energy and nanocrystalline metals nucleate at grain boundaries, triple junctions or twin intersections [26–34]. Based on these observations, it is generally assumed that voids in all pure metals initiate at these features [35]. However, the recent work of Boyce *et al.* [36] suggests that the dislocation boundaries created during deformation may also act as important sites for void nucleation. This hypothesis is supported by early investigations of void initiation in pure, single crystalline metals by Wilsdorf and coworkers. These studies, summarized in Wilsdorf's seminal 1983 review [25], established that, in the absence of particles and grain boundaries, void initiation occurs at deformation-induced dislocation boundaries. An example of this is presented Fig. 1. Jagannadham *et al.* [37] hypothesized that these voids nucleated to relieve the strain energy associated with the deformation-induced boundary after a critical boundary misorientation was reached. However, the case of void initiation in a pure, bulk, polycrystalline material has not been extensively investigated. This study thus examines if deformation-

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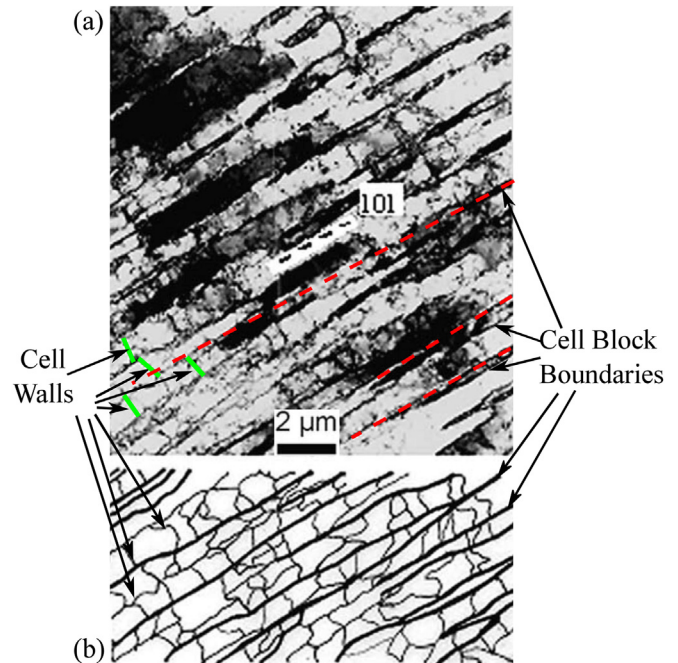
E-mail address: [pnoell@sandia.gov](mailto:pnoell@sandia.gov) (P. Noell).



**Fig. 1.** The black arrows in this transmission electron microscope (TEM) micrograph mark voids that initiated at a deformation-induced dislocation boundary in a strained Be single crystal. This image is from Gardner *et al.* [38].

induced boundaries or grain boundaries are the primary site for void nucleation in pure, high-stacking fault energy metals, specifically tantalum.

Beginning with the research of Hansen and Kuhlmann-Wilsdorf in the early 1990's [39], work over the past 25 years demonstrated that two distinct kinds of deformation-induced boundaries exist: dislocation cell walls and cell block boundaries (these boundaries can also be described as incidental and geometrically necessary boundaries using the terminology of Kuhlmann-Wilsdorf) [40]. Examples of both of these are illustrated in Fig. 2. Dislocation cell walls delineate small (0.5–2 μm), equiaxed volume elements (dislocation cells) and generally have misorientations of less than 2° [41]. Dislocation cells are typically organized into cell blocks: extended, planar features that often run parallel to the trace of a highly stressed slip system [42]. Because both kinds of deformation-induced boundaries generally form during room-temperature deformation, publications before that of Kuhlmann-Wilsdorf *et al.* [39] in 1991, including Wilsdorf [25], generally referred to deformation-induced boundaries as dislocation cell boundaries, cell walls or subgrain boundaries and these terms are still used to refer generally to deformation-induced boundaries. However, important distinctions exist between cell walls and cell block boundaries. For example, cell block boundaries generally have significantly higher misorientation angles than cell walls. In addition, while slip activity within all the dislocation cells in a



**Fig. 2.** (a) A TEM micrograph and (b) a sketch of the microstructure in a grain of an interstitial-free steel (a BCC metal) specimen reduced 10% by cold-rolling are shown. Cell block boundaries are marked in bold on the sketch in (b). These cell blocks are subdivided by dislocation cell walls, which are marked in light gray in the sketch in (b). This image is from Li *et al.* [42].

single cell block is generally similar, different slip systems are active in neighboring cell blocks [41]. As a result, the misorientation angle across a cell block boundary increases with increasing strain, but the misorientation angle across cell walls does not vary significantly with strain (for strains greater than approximately 0.5 [40,43–45]). If relieving the stored strain energy associated with deformation-induced boundaries is the primary mechanism for void initiation in pure metals, it is hypothesized that void initiation at deformation-induced boundaries will primarily occur at (evolving) cell block boundaries rather than (stagnant) cell walls. This study examines this hypothesis by investigating where voids initiate in polycrystalline tantalum following quasistatic tensile deformation.

To collect statistically-significant evidence of void nucleation in tantalum, scanning electron microscopy (SEM) was used to identify dozens of incipient voids in mid-plane (see Fig. 3) cross-sections of deformed, polycrystalline tantalum tensile samples. By examining dozens of voids rather than just a few as with past work [33,36], it is possible to form a more global perspective on the spectrum of microstructural conditions associated with void nucleation. Electron backscatter diffraction (EBSD), transmission Kikuchi diffraction (TKD) and transmission electron microscopy (TEM) were subsequently used to characterize the microstructure around these voids to answer the following questions:

- what are the microstructural feature(s) associated with quasi-static, room temperature void nucleation in polycrystalline tantalum?
- what commonalities do these microstructural features share that make them the preferred sites for void nucleation?

The answers to these questions provide an important first step towards understanding void nucleation and developing a mechanistically-based model for void nucleation in pure,

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