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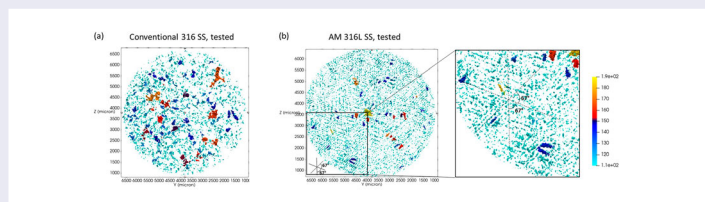
## Understanding creep behaviors of additively manufactured 316L stainless steel via void characterization

Xuan Zhang<sup>a</sup>, Christopher P. Carter<sup>a\*</sup>, Yashas Satapathy<sup>a\*\*</sup>, Aniket Tekawade<sup>b,\*\*\*</sup>, Jun-Sang Park<sup>c</sup>, Peter Kenesei<sup>c</sup> and Meimei Li<sup>a</sup>

<sup>a</sup>Nuclear Science and Engineering Division, Argonne National Laboratory, Lemont, IL, USA; <sup>b</sup>Data Science and Learning Division, Argonne National Laboratory, Lemont, IL, USA; <sup>c</sup>X-ray Science Division, Argonne National Laboratory, Lemont, IL, USA

### ABSTRACT

Additively manufactured (AM) 316L stainless steel (SS) has been reported to have low creep resistance compared to its conventionally made counterparts. Herein, we quantitatively characterized the voids in a creep-ruptured AM 316L SS specimen and those in a conventional specimen ruptured under the same testing condition. The AM 316L SS contained more small creep voids and fewer large ones. 3D reconstructions showed the spatial distribution of the small voids in AM 316L SS followed the melt pool tracks, which was attributed to the grain structure unique to the laser process. The observations explained the creep behaviors of the two specimens.



### IMPACT STATEMENT

This paper reports that the grain structure resulted from the laser printing process has critical effects on the creep void formation in AM 316L SS.

### ARTICLE HISTORY

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### KEYWORDS

Laser powder bed fusion; x-ray tomography; electron backscattering diffraction; stainless steel; creep

Type 316 austenitic stainless steel has been widely used or considered in various types of nuclear reactors, including the current light water reactors and the next-generation advanced reactor concepts. Metal additive manufacturing (AM) technologies such as laser powder bed fusion (LPBF) have the potential to transform the nuclear industry by producing high quality, custom-designed components faster and cheaper with enhanced properties, thus helping with extending the operating life and enhancing the performance of current plants while also reducing the cost and improving the performance of advanced reactor concepts [1]. Previous studies have shown that AM 316L stainless steels have considerably improved low-temperature tensile strength and good ductility over conventionally fabricated wrought 316 SS [2–5]. However, more information is needed to assess its application in advanced reactors, in which high-temperature time-dependent and cyclic properties,

e.g. creep, fatigue, creep-fatigue, must be thoroughly investigated. A database containing that information must be built for the material to qualify its use.

Data on the short-term creep performance of AM 316L SS have indicated that the material has inferior creep resistance over the conventional 316 SS [6–8]. Li et al. [6] showed that the AM 316L SS had a shorter creep life but a comparable creep ductility when compared to the literature data on conventional 316 SS. Optical microscopy was employed in that study to show that the creep fracture was intergranular at the test conditions. The shortened duration of the secondary creep stage and the early onset of the tertiary creep stage in AM 316L was explained by the microstructural recovery and the existence of built-in voids, and the comparable ductility between the AM 316L SS and the conventional 316 SS was suggested to be due to the reduced likelihood of carbide precipitation, a higher density of microcracks, and the

**CONTACT** Xuan Zhang ✉ [xuanzhang@anl.gov](mailto:xuanzhang@anl.gov) Nuclear Science and Engineering Division, Argonne National Laboratory, Lemont, IL 60439, USA

\*Present address: Department of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139, USA

\*\*Present address: Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC 27695, USA

\*\*\*Present address: Picarro, Inc., Santa Clara, CA 95054, USA

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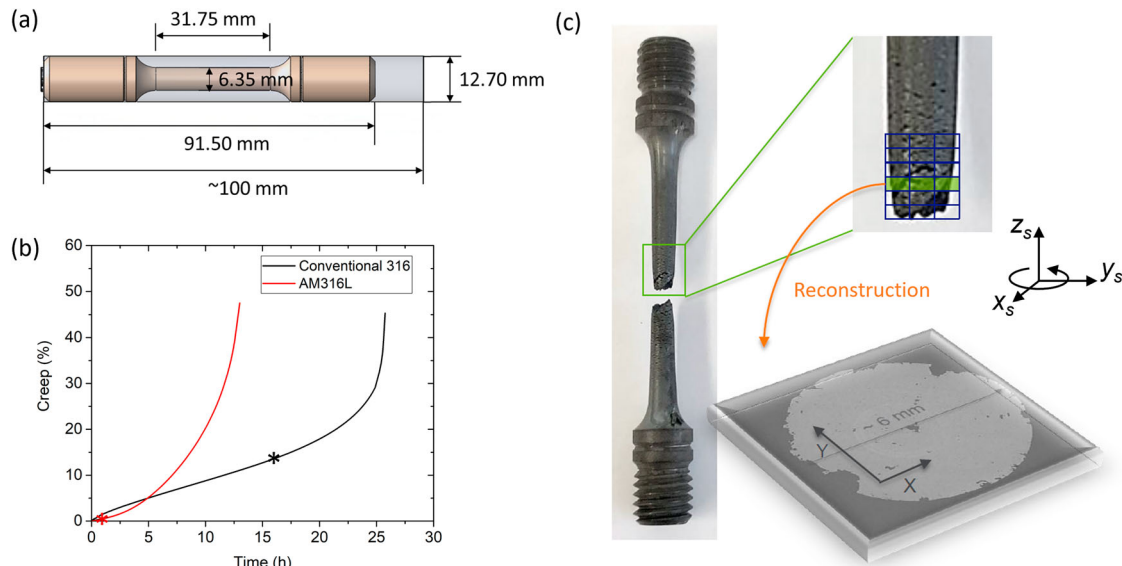


**Figure 1.** (a) Geometry of the untested creep specimen manufactured from an AM rod. (b) Creep curves of AM 316L and conventional 316 SS specimens tested at 650°C, 225 MPa; the onset of the tertiary stage on each curve was marked with an asterisk. (c) Illustration of the stitching tomography of the creep-tested AM 316L specimen and a slice from the reconstruction showing the gauge cross section near the fracture surface.

unique, layered grain structure in AM 316L SS [6]. However, direct microstructural evidence was lacking. In the present study, high-energy synchrotron x-ray computed tomography (CT) and electron-backscatter diffraction (EBSD) techniques were employed to provide further insights into the creep behavior of AM 316L SS. The void population in volumes that are directly underneath the fracture surfaces in a creep-ruptured AM 316L SS specimen and a creep-ruptured conventional 316 SS specimen were characterized. The void population in an untested AM 316L SS specimen was also characterized for comparison. The AM 316L SS creep specimen was fabricated in the as-built condition from an AM 316L SS rod that was nominally 0.5 in. (12.7 mm) in diameter and 4 in. (101.6 mm) in height (Figure 1(a)) from a build manufactured by Oak Ridge National Laboratory (ORNL) [9]. The build was printed by LPBF using a Concept Laser-M2 printer, using a stripe scan strategy, a laser hatch spacing of 90  $\mu\text{m}$ , a laser spot size of 130  $\mu\text{m}$  and a rotation angle of 67° (private communication with ORNL). The conventional 316 SS specimen was machined out of a plate that was hot rolled and solution annealed. The gauge length direction was the rolling direction.

For the AM 316L specimen, the axial direction is the build direction. The creep tests were performed in air at 650°C and 225 MPa. The creep curves are shown in Figure 1(b). The creep rupture life for the AM 316L SS specimen was 13 h, while that for the conventional 316 SS was 25 h. In contrast to the conventional 316 SS specimen which showed an extended steady-state creep stage, the AM 316L SS specimen showed minimal steady-state

creep. The time to the onset of tertiary creep (determined by the intersection of the creep curve with a straight line that is 0.2% above a fitted line to the steady-state creep stage [10]) was 1 h for the AM specimen and 16 h for the conventional specimen. The total elongation was 47.5% for the AM specimen and 45.3% for the conventional specimen.

High-energy synchrotron x-ray CT was performed at the beamline 1-ID of the Advanced Photon Source (APS) at Argonne National Laboratory (ANL). Three specimens were measured, including the creep-tested AM 316L, the creep-tested conventional 316 SS, and one untested AM 316L. The x-ray energy was 80.725 keV and the beam size was 2.2 mm (width)  $\times$  1.0 mm (height). The nominal detector pixel size was 1.172  $\mu\text{m}$ . For the untested AM 316L specimen, one layer was measured around the center of the gage region. For the creep-tested AM 316L and conventional 316 specimens, the measurements started from the fracture tip and a total of 6 consecutive layers for each specimen were measured. Since the nominal diameter of those specimens ( $\sim 6$  mm) were much larger than the beam width, a total of three field-of-views (FOVs) were needed to cover the entire cross-section of a specimen in one layer, as seen in Figure 1(c). Such a measurement was commonly named ‘stitching tomography’ [11]. As a result, for each creep specimen, a total of 18 FOVs were measured, totaling  $\sim 450$  GB in raw data size.

Such a large data size is beyond the processing capability of a common desktop computer for reconstruction and analysis. First, we utilized a workstation to run a CT reconstruction code developed by the beamline and

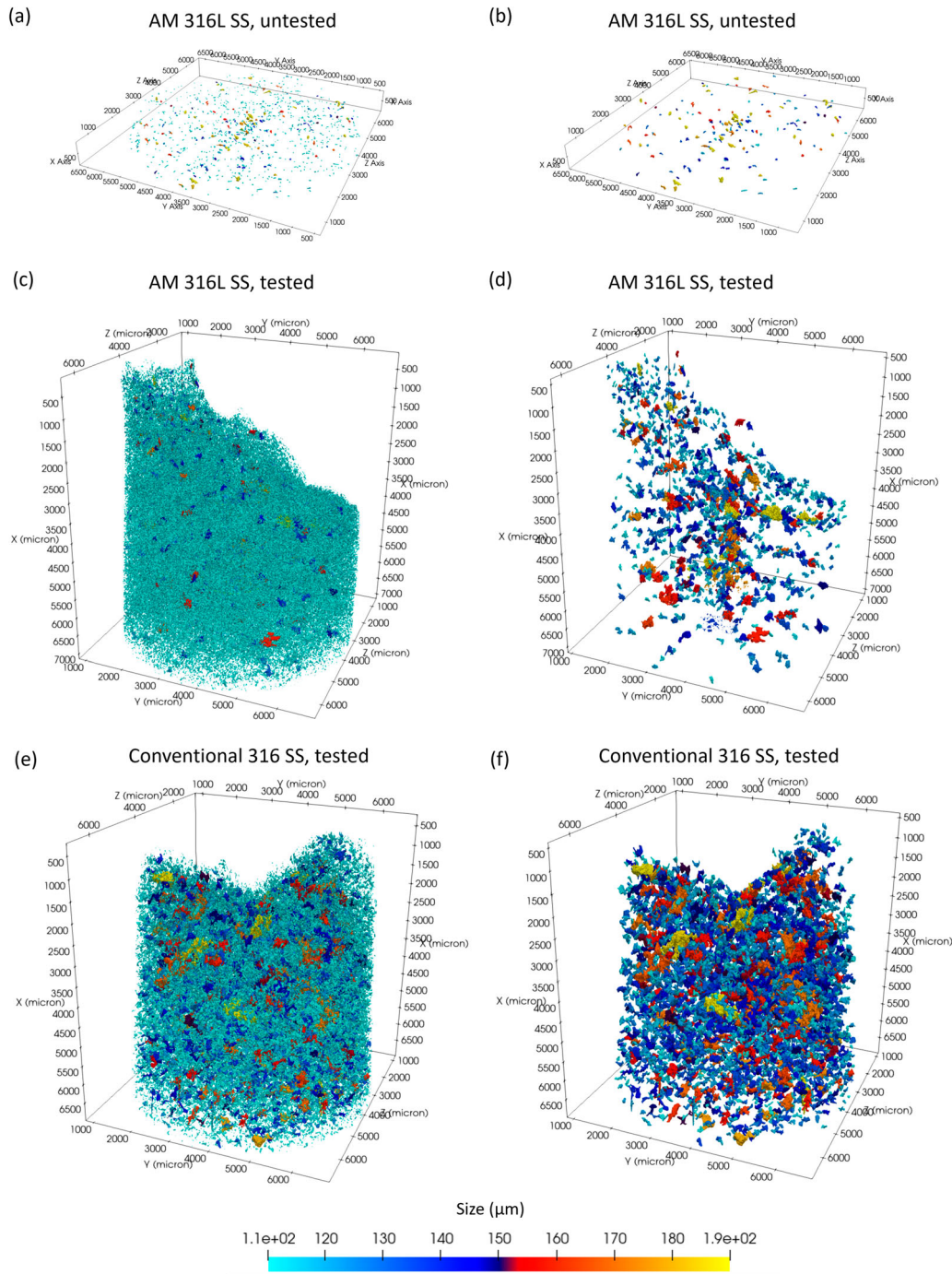


**Figure 2.** Visualization of the voids in the creep-tested AM 316L SS and the conventional 316 SS specimens: (a) all voids in untested AM 316L SS; (b) voids larger than 110  $\mu\text{m}$  in untested AM 316L SS; (c) all voids in creep-tested AM 316L SS; (d) voids larger than 110  $\mu\text{m}$  in creep-tested AM 316L SS; (e) all voids in conventional 316 SS; (f) voids larger than 110  $\mu\text{m}$  in conventional 316 SS.

obtained the stitched reconstruction slices of each layer. Then, the reconstruction slices were fed into a new open-source code ‘Tomo2Mesh’ developed at ANL [12,13] for feature extraction and analysis. The code utilized a compact 3D convolutional neural network to perform denoising and binarization, and then adopted a voxel subset reconstruction scheme to first identify locations where the voids might exist by a coarse mapping scheme, and

once the existences were confirmed, the code recovered the full voxel resolution of the voids. Such an approach required the input of a threshold size, which involved checking each dimension (Xs, Ys, Zs) of the void and seeing if any of these values were less than or equal to the threshold value. If so, the void would not be reconstructed. In this study, a threshold of 10  $\mu\text{m}$  was applied. The algorithm together with the utilization of

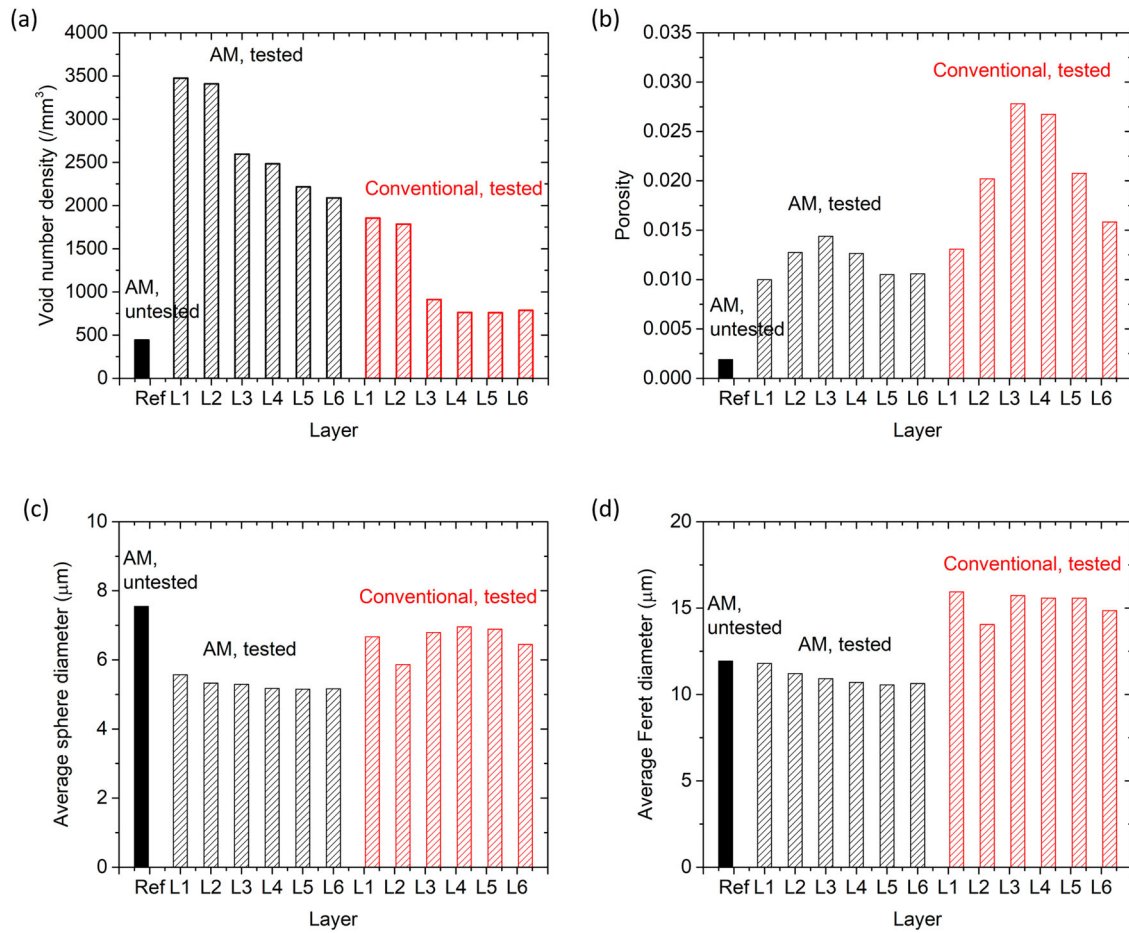


**Figure 3.** Plots of the statistical data regarding the voids in each of the measured volumes in the untested AM316L SS, the creep-tested AM 316L SS, and the creep-tested conventional 316 SS specimens: (a) the void number density; (b) the porosity; (c) the average sphere diameter; (d) the average maximum Feret diameter. In each of the tested specimens, Layer 1 (L1) is closest to the fracture tip and Layer 6 (L6) is farthest away.

GPU computing led to a significant reduction of net computing time from hours to minutes.

The Tomo2Mesh code outputs surface meshes of the voids into a .ply file which were imported into Paraview software for visualization [14]. The code also outputs statistical results of feature analysis. Figure 2 shows the visualizations of the voids in one layer of the untested (as-built) specimen, and the fracture tip areas of the creep-tested AM 316L and conventional 316 SS specimens. For each specimen, two figures are shown; the ones (Figure 2(a,c,e)) with a much higher void density show all the reconstructed voids ( $> 10 \mu\text{m}$ ), while the ones (Figure 2(b,d,f)) with a lower density only show the voids with a sphere-equivalent diameter (i.e. diameter of a sphere that has the same volume as the void) larger than  $110 \mu\text{m}$ . The color range was tuned to highlight the larger voids. Statistics of the voids in those three specimens are presented in Figure 3. Note that the voids presented in this study exclude any surface cracks. In Figure 3, for the tested specimens, Layer 1 (L1) is closest to the fracture tip

and Layer 6 (L6) is farthest away. It is seen that the void number density (i.e. number of voids per unit volume) and the porosity level (i.e. the volume of reconstructed voids divided by the volume of material) in the untested AM 316L SS specimen ( $460/\text{mm}^3$  and  $0.002$ , respectively) is only a small fraction of those in the two tested specimens. The void number density in each tested specimen decreases as the measured volume moving away from the fracture surface area (from L1 to L6) which agrees with the fact that most voids are located near the fracture surface. The void number density in the AM specimen ( $\sim 2500/\text{mm}^3$ ) roughly doubles that in the conventional specimen ( $\sim 1000/\text{mm}^3$ ), but the porosity is about half ( $\sim 0.01$  in AM 316L vs  $\sim 0.02$  in conventional 316). The average sphere-equivalent diameter and the average maximum Feret diameter in the AM 316L SS ( $\sim 5$  and  $11 \mu\text{m}$ , respectively) are much smaller compared to those in the conventional 316 SS ( $\sim 7$  and  $15 \mu\text{m}$ , respectively). Such information, combined with the observations from Figure 2, confirms that the AM 316L SS has a much

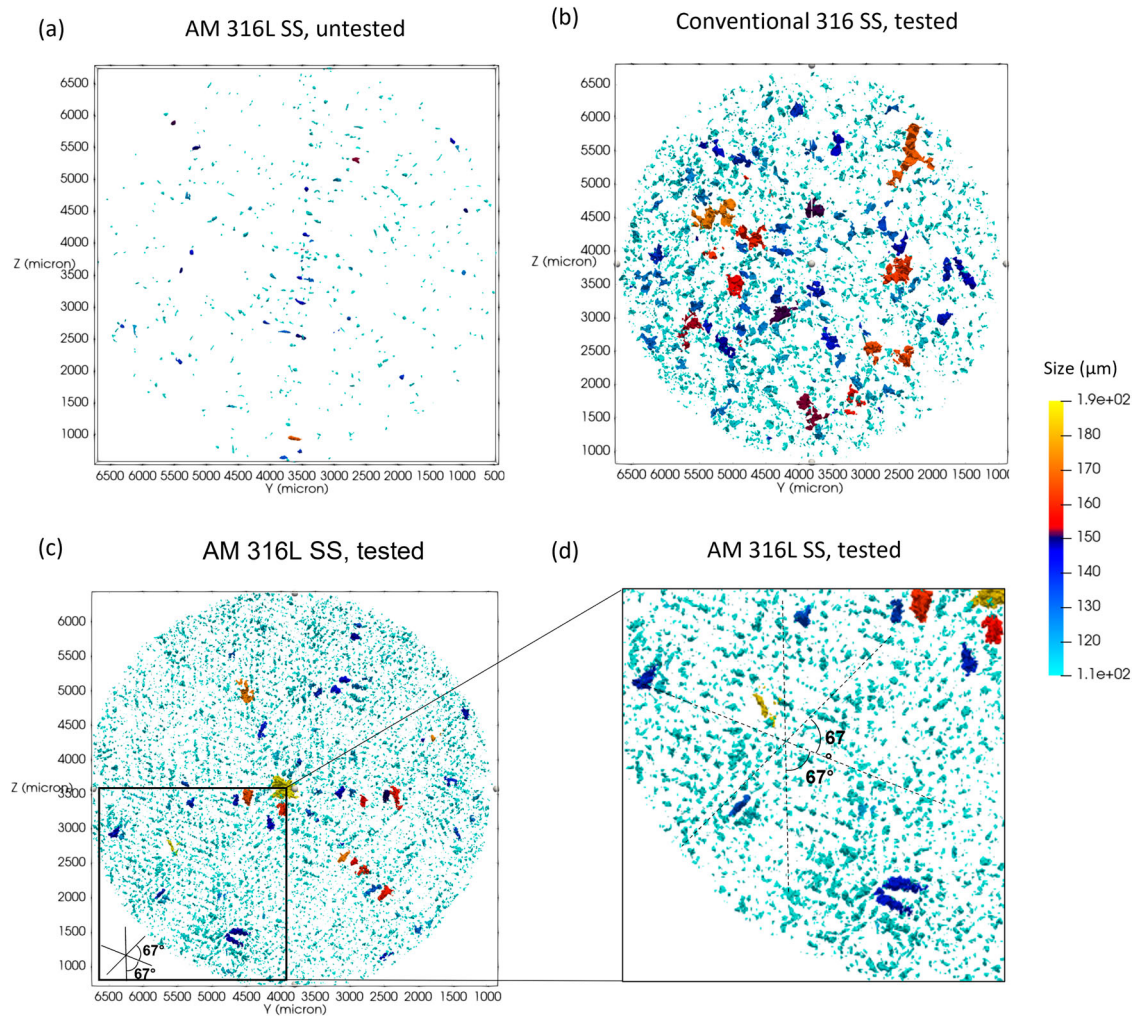


**Figure 4.** (a–c) Visualization of the voids in a 0.2-mm thick slab perpendicular to the loading direction near the fracture tip of (a) the untested AM 316L specimen, (b) the creep tested conventional 316 SS specimen, (c) the creep tested AM 316L specimen, and (d) a closer view of an area in (c) showing the alignment of the small voids into line patterns.

higher density of small voids and a much lower density of large voids compared to the conventional 316 SS in the fracture tip areas.

The visualizations of all the voids in a 0.2-mm slab perpendicular to  $Z_s$  in each specimen are shown in Figure 4. It is observed that the voids in the untested AM 316L SS specimen tend to align in the radial directions forming a spoke-like structure. For each of the tested specimens, the presented volume is from the middle of Layer 4 (L4), which is about 3.5 mm away from the fracture tip. The visualization of the AM 316L specimen (Figure 4(c)) reveals that the larger voids are arranged into the spoke-like structure, suggesting that they were the built-in voids which grew larger with deformation. On the other hand, numerous small voids are observed to arranged into line patterns that can be decomposed into three sets of parallel lines rotating by  $67^\circ$  from each other (see the illustrations in Figure 4(c and d)).  $67^\circ$  was the rotating angle of the hatch patterns between adjacent layers in the laser

printing of this material. The spacings between the parallel lines are nominally  $90\ \mu\text{m}$ , also matching the laser hatch spacing of  $90\ \mu\text{m}$ . This observation indicates that the laser scan path has a critical role in the void formation in this AM material. In contrast, Figure 4(b) shows that the voids in the conventional material are randomly distributed.

Further insights were obtained with the EBSD technique. The EBSD mapping was performed with a JEOL IT800HL SEM with an electron energy of 15 keV.  $10^\circ$  was used to identify high-angle grain boundaries. The EBSD map of an area in the untested conventional 316 SS is shown in Figure 5(a). The area-weighted average mean of the equivalent circle diameters is  $231 \pm 85\ \mu\text{m}$ . The EBSD map of the untested grain structure in the normal-to-build plane is shown in Figure 5(b). The schematics of the melt pools are superimposed in Figure 5(b), based on the observations that the grains at the centers of the melt pools are much finer and more equiaxed compared

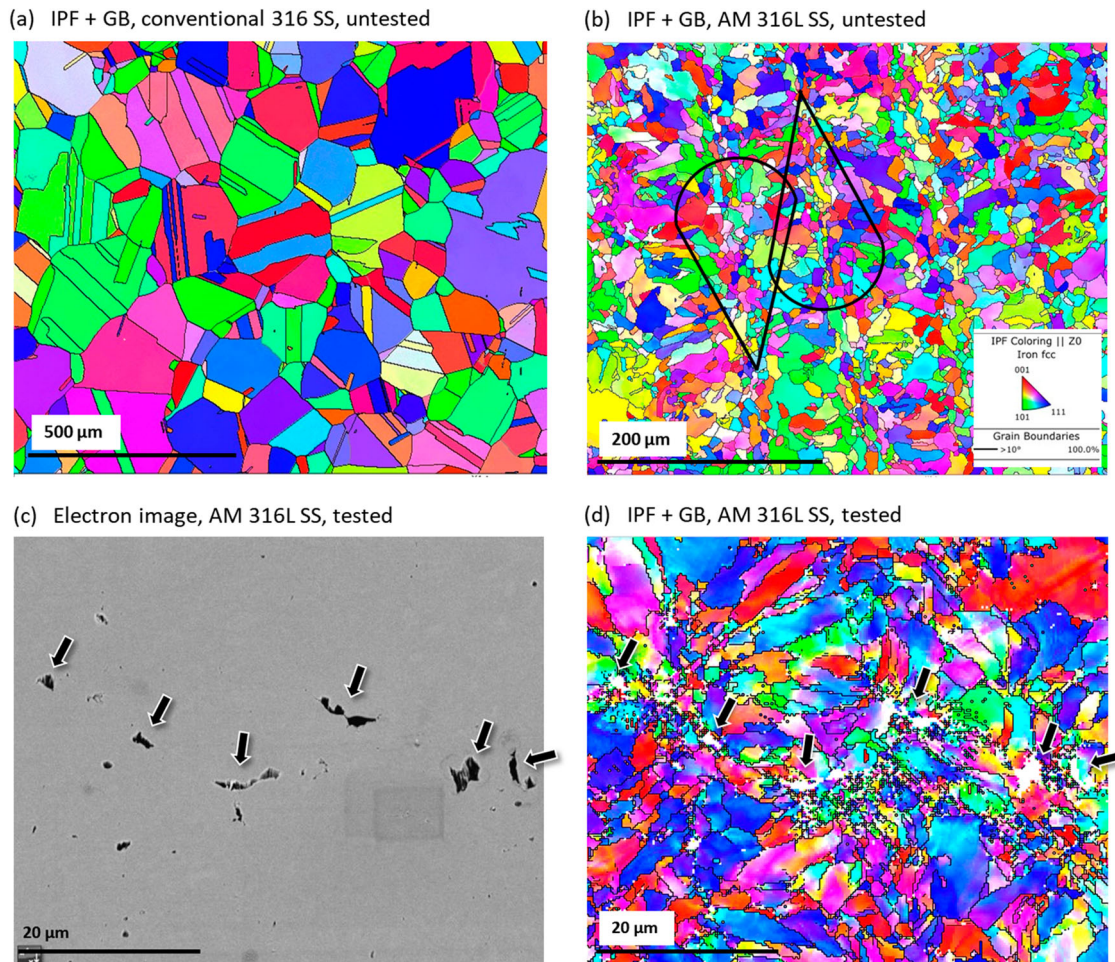


**Figure 5.** (a) The EBSD Inverse Pole Figure (IPF) + Grain Boundary (GB) map of an area in a untested conventional 316 SS specimen; (b) The EBSD IPF + GB map of an area normal to build in a untested AM 316L SS specimen; (c) The secondary electron image of a cross-sectional area close to the fracture tip of the creep-tested AM 316L SS specimen; (d) The EBSD IPF + GB map of the same area as in (c). The arrows in (c) and (d) highlight the intergranular cracks.

to those near the fusion lines, similar to Zhou et al. [15] and Andreau et al. [16]. is due to the rapid solidification of the melt pool that favors an epitaxial growth of grains whose orientations are controlled by the crystals already present, and therefore, coarse, elongated grains are formed on the sides of the melt pool tracks while fine, equiaxed grains are formed near the center of the melt pool tracks. In other words, the grain structure possesses clear trace of the laser path. The area-weighted average mean of the equivalent circle diameters of all the grains is  $31 \pm 8 \mu\text{m}$ ; that of the grains at the centers of the melt pools is  $14 \pm 5 \mu\text{m}$  while that of the grains on the sides of the melt pools is  $40 \pm 11 \mu\text{m}$ . After the creep test, EBSD maps of cross-sectional areas close to the fracture tip show that the small voids detected in x-ray CT are intergranular microcracks. Intergranular microcracking has also been confirmed in a previous metallographic study of the same samples in regions near the fracture surfaces [6]. EBSD maps also show that the microcracks

are predominantly formed within the fine-grain zones or at the intersections of fine-grain zones and coarse-grain zones; Figure 5(c and d) show an example. Such observations suggest that the microcracks are formed preferably at the centers of the melt pools where the fine grains are located, and therefore, their spatial distribution depict the laser hatch pattern, as observed by x-ray CT (Figure 4(c and d)).

Li et al. [6] attributed the shortened secondary creep stage (below 1% strain and for  $\sim 1$  h) to the microstructure instability, primarily to the recovery of the dislocation cell structure upon the onset of creep deformation as evidenced by transmission electron microscopy (TEM) observations, and the shortened creep rupture life to the built-in porosity serving as nucleation sites of creep voids. The present work provides new insight by examining the grain structure and the void distribution. The grains are much finer in the AM material ( $31 \mu\text{m}$ ) compared to the conventional material

(231  $\mu\text{m}$ ), and the grains in the center of the melt pool tracks (14  $\mu\text{m}$ ) are finer compared to those on the sides (40  $\mu\text{m}$ ). It is known that the void formation is much easier in the fine-grain material compared to the coarse-grain material due to the higher grain boundary area [17], which explains the observations that (1) there are more small voids at the grain boundaries of the creep-tested AM 316L compared to those in the coarse-grained conventional material, and (2) those small voids in the tested AM material are spatially arranged along the centers of the laser tracks where the grains are finest. Creep voids can directly lead to the tertiary creep stage, thus providing an explanation to the early onset of the tertiary stage in the AM material. The small voids in the tested AM material were either newly formed from the creep deformation, or they grew from built-in voids that initially fell under the x-ray CT detection limit. However, the likelihood of the latter is low from SEM studies of the untested material (results not presented), which did not reveal the existence of many sub- $\mu\text{m}$  or a few  $\mu\text{m}$  sized built-in voids along grain boundaries. On the other hand, the crack propagation was hindered by the higher intergranular void/microcrack density resulted from the finer grain sizes [18–20]. The rationale is that the grain boundary triple junctions of certain types can effectively arrest the microcracks and prevent them from coalescence. Since finer grains possess more triple junctions, the microcracks are in general shorter in fine-grained materials compared to coarse-grained materials. This explains the overall comparable or extended period of the tertiary creep stage and the comparable creep ductility in AM 316L compared to the conventional material (Figure 1(b)). The original built-in voids in AM 316L that are arranged into spoke patterns grew larger during the test and became the largest voids in the fractured specimen. Little evidence of newly formed large voids (i.e. large voids formed entirely during the creep test) is observed, suggesting that the original built-in voids have a critical role in the final rupture of the AM specimen when large voids/cracks coalesce.

In summary, the void distribution near the fracture tip area in a creep-tested AM 316L specimen was characterized by high-energy synchrotron x-ray CT and SEM-EBSD and was compared to a conventional 316 SS specimen tested under the same condition. Both specimens failed by intergranular cracking. It was discovered that for the crack tip areas, the AM specimen contains more small voids and fewer large voids than the conventional 316 SS. The small voids tend to form along the laser tracks, preferably at the centers of the melt pools where fine grains are located. Those small voids in the AM specimen had a critical role in accelerating the onset of the tertiary creep stage, extending the duration of the

tertiary creep stage and maintaining a high ductility in the creep deformation. The built-in voids grew into the largest voids in the tested AM specimen, which could be directly responsible for the final rupture. The results from this study suggest that optimizing the grain structure while maintaining a low as-build porosity is a key to increase the creep resistance of AM materials.

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## Disclosure statement

No potential conflict of interest was reported by the author(s).

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