# Cyclic response of additive manufactured 316L stainless steel: The role of cell structures 

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#### Abstract

We report the effect of cell structures on the fatigue behavior of additively manufactured (AM) 316L stainless steel (316LSS). Compared with the cell-free samples, the fatigue process of fully cellular samples only consists of steady and overload stages, without an initial softening stage. Moreover, the fully cellular sample possesses higher strength, lower cyclic softening rate and longer lifetime. Microscopic analyses show no difference in grain orientations, dimensions, and shapes. However, the fully cellular samples show planar dislocation structures, whereas the cell-free samples display wavy dislocation structures. The existence of cell structures promotes the activation of planar slip, delays strain localization, and ultimately enhances the fatigue performance of AM 316LSS. © 2021 The Author(s). Published by Elsevier Ltd on behalf of Acta Materialia Inc.


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Additive manufacturing with its unique advantages of design freedom and rapid manufacturing capability has gained huge scientific interest [1,2]. Among the alloys fabricated by additive manufacturing, 316L stainless steel (316LSS) with face-centered cubic (FCC) structure has attracted huge attentions for its wide application range and the unprecedented mechanical properties [3,4], e.g., the yield strength of 552 MPa and failure elongation of $83.2 \%$ was achieved for the AM 316LSS [1], whereas the wrought-annealed sample shows a yield strength of 244 MPa and elongation to failure of $63 \%$ [5]. The superior tensile strength is primarily attributed to the notable impediment effect for dislocation motion from the stable cell structures [1], and the Hall-Petch typed strengthening behavior, namely the yield strength scaling with the cell size, is usually applied to rationalize the high strength [2,6]; while the large elongation correlates to the steady and progressive workhardening mechanism provided by the prolific and complicated interactions between dislocations and cell structures [2]. Therefore, the cell structures play a vital role in the outstanding tensile performance of AM 316LSS. To date, extensive studies mainly reveal the monotonic deformation behavior of AM alloys [7-9]. However,

[^0]engineering materials are usually loaded cyclically not monotonically in the practical services. As we reviewed the previous reports about AM alloys, the data on fatigue properties are very limited [10], especially the effects of cells on the fatigue behavior (such as cyclic stress/strain responses), even if these cells were proved very common in AM alloys [11]. Recently, Elangeswaran et al. [12] revealed much better fatigue properties of the as-built AM 316LSS compared to the conventionally manufactured counterparts, which was considered to be caused by the strong resistance of cellular structures to crack initiation. However, to our knowledge, investigations concerning the cell structure effects on the fatigue behavior of AM alloys, especially considering dislocation-based deformation mechanisms, are at the forefront of development but little is yet known.

It is known that the fatigue behavior of FCC materials is closely related to the dislocation slip modes, i.e., plane slip or wave slip [13]. Typical dislocation substructures in fatigue microstructures include planar slip bands (SBs), stacking faults (SFs), deformation twins (DTs) as well as dislocation cells/veins and persistent slip bands (PSBs). Among them, SBs, SFs and DTs are prominent dislocation substructures in FCC materials with low stacking fault energy (SFE) [14,15], while dislocation cells/veins and PSBs are usually formed in materials with high SFE [16-20]. PSBs are composed of prefect edge dislocations with the same Burgers vector,
and usually formed in single-slip orientated grains [16,18,19,21]. On the contrary, the formation of dislocation cells/veins is caused by cross-slip of prefect screw dislocations [20], and illustrates the activation of secondary slip systems [13,19]. DTs are effective strengthening substructures in metals [22], and their formation requires high stress concentrations [1,22]. The cell structure decorated with elemental segregations and tangled dislocations, is the most significant microstructural feature in AM alloys, and has never been found in bulk materials fabricated by any other manufacturing methods. Therefore, the present study aims to identify the fatigue behavior and related microstructure evolution of AM 316L SS with different concentrations of cell structures, and to determine whether the observed differences are driven by the cell structures.

316LSS cylindrical bars with a height of 70 mm and a diameter of 10 mm were vertically fabricated in an argon environment by a commercial EOSINT M280 machine with a maximum 200 W fiber laser. The initial gas-atomized 316L SS powders have a particle size distribution ranging from 15 to $50 \mu \mathrm{~m}$. The actual chemical composition of the powders and the adopted optimal printing parameters during the AM process have been reported in our previous work [3]. In order to obtain microstructures with different volume fractions of cell structures, various heat treatments were conducted on the as-built bars, namely annealed at 900 and $1050{ }^{\circ} \mathrm{C}$ for 10 min and followed by water quench. Subsequently the heat-treated bars were machined into the fatigue specimens with a gauge dimension of 15 mm in length and 5 mm in diameter. For simplicity, we named the specimens with the annealing conditions, e.g., 900 ${ }^{\circ} \mathrm{C}-10$ min represents the as-built sample annealed at $900^{\circ} \mathrm{C}$ for 10 min.

The load-controlled fatigue tests were performed on an MTS servo-hydraulic fatigue test rig at room temperature using total stress ranges from 673 MPa to 793 MPa . The targeted stress range was adjusted after each test in order to cover the range of $10^{4}$ to $10^{6}$ cycles to failure as good as possible with the few numbers of samples available. A sinusoidal stress waveform ( $\mathrm{R}=-1$ ) was employed at a constant frequency of 5 Hz . The corresponding strain response was measured by an Instron extensometer with a gauge length of 12.5 mm . To reveal the microstructural evolution, microscopy analyses via scanning electron microscope (SEM), electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM) were conducted on the specimens before and after fatigue testing. For EBSD, a Hitachi SU70 FEG SEM equipped with a Nordlys-S ${ }^{\mathrm{TM}}$ detector was employed with a step size of 200 nm and a binning of $2 \times 2$. Small blocks parallel to the loading direction were extracted from the gauge section of the fractured samples, and then mounted, ground and polished according to the Struers recommendations. The acquired EBSD data were analyzed using Channel 5 and the MTEX software. TEM samples were extracted near the crack initiation sites, and mechanically ground to a thickness of $\sim 60 \mu \mathrm{~m}$. Thereafter, the foils were twinjet electro-polished in a solution of $10 \mathrm{vol} \%$ perchloric acid and $90 \mathrm{vol} \%$ ethanol at 25 V and $-25^{\circ} \mathrm{C}$. TEM investigations were performed on an FEI Tecnai G2 microscope operating at 200 kV .

The total stain range as a function of cycle number for the three batches of samples under the similar cyclic stress are presented in Fig. 1a-1c. Similar evolution also can be found under other fatigue conditions (see the supplementary material S 1 ). The strain response of the heat-treated samples in Fig. 1b and c can be divided into three stages: an initial increase (cyclic softening stage), followed by a minor change (steady stage), and finally by a dramatically increase until failure (overload stage). The initial softening stage occurs in the first 200 to 500 cycles (about $1.8 \%$ lifetime in Fig. 1e), while the following steady stage occupies more than $95 \%$ of lifetime, illustrating that the majority of fatigue process is spent in the steady stage. Interestingly, as shown in Fig. 1a, the
fatigue process of the as-built sample only consists of the steady and overload stages, which will be analyzed in detail later. Moreover, the as-built sample exhibits notable lower cyclic softening rate than the annealed ones (Fig. 1d). For example, under the normalized cycle number of 0.8 , the as-built sample exhibits a total strain range of $0.5 \%$, which is approximately two fifths the strain of the $1050^{\circ} \mathrm{C}-10 \mathrm{~min}$ sample. The lower strain range in the asbuilt sample is caused by its higher cyclic/yield strength with the nearly same elastic modulus (Fig. 1f). Higher yield strength of asbuilt samples has been frequently reported [7], which is due to the significant strengthening caused by numerous dislocations generated during the ultrafast cooling process [3]. Fig. 1g presents the plot of total stress range $\left(\Delta \sigma_{T}\right)$ versus the cycles to failure $\left(\mathrm{N}_{\mathrm{f}}\right)$. For a given $\Delta \sigma_{T}$, the as-built sample possesses a much longer fatigue life than the heat-treated ones.

Fig. 2a and b show representative overlaid back contrast (BC) and orientation maps of the as-built and $1050^{\circ} \mathrm{C}-10$ min samples acquired via EBSD parallel to the building direction (BD). Both alloys possess columnar grains with similar grain orientation and an average grain size of about $19.7 \mu \mathrm{~m}$. Further increasing the magnification, as illustrated in Fig. 2c-d, the microstructure of the asbuilt sample is decorated by full cell structures while the $1050^{\circ} \mathrm{C}$ 10 min sample is free of cells, which was also confirmed by the TEM images (Fig. 2e-f), showing that dislocations are randomly distributed after heat treatment. Therefore, the only difference in the microstructure features of these two samples is the existence of cell structures or not, and we can use them to study the mechanisms of cells on the cyclic response behavior of AM alloys.

Fig. 2 g and h provide representative overlaid BC and orientation maps as well as KAM (kernel average misorientation) maps of the fatigued as-built and $1050^{\circ} \mathrm{C}-10$ min samples, respectively. Compared with the undeformed state (Fig. 2a and b), the orientation maps illustrate no distinct changes in crystallographic orientations and dimensions of the grains. However, large population of SBs were formed in the grain interiors and intersected with grain boundaries (GBs), causing significant stress concentrations along SBs (indicated by yellow arrow in Fig. 2g2) and GBs (indicated by red arrows in Fig. 2g2), and may eventually lead to the microcrack initiation. A more detailed analysis of the crack source and fracture behavior of the present 316LSS can be found in supplementary material S2. Furthermore, some DTs were formed along the SBs in the as-built sample (Fig. 2 g 1 ), which are not readily available in conventional counterparts [23,24]. DTs have significant influences on dislocation motion and delay strain localization [22]. From Fig. 2h1, it can be seen that DTs are not present in the $1050^{\circ} \mathrm{C}-10 \mathrm{~min}$ sample. By ruling out the SFE effect (Section S3 in the Supplementary material), it can be speculated that the different behavior in DT formation of the two samples is caused by cell structures. In other words, the cell structures promote twinning for AM 316LSS during plastic deformation.

In deformed as-built samples, typical dislocation substructures include SBs, SFs and DTs (Fig. 3). Among them, the SFs and SBs, along with their debris, are the most dominant defect structures, while DTs only can be observed within the region 3 mm from the fracture. The SBs are composed of parallel dislocations (Fig. 3a and b), which agrees well with the observations in Ref. [25]. In some grains, DTs are extended in different orientations, and significant interactions with dislocations, cell structures and other twins also occurred, as revealed in Fig. 3e and h. Besides, with the cyclic stress range increasing, the density of DTs and their interactions with other dislocation substructures also notably increase, which may be caused by the severer heterogeneous deformation under high cyclic stress. It is known that twins are planar defects and comprised of at least two adjacent SFs [26]. From the enlarged images in Fig. 3d and $g$, it can be seen that all SFs originate from cell walls, which verifies the aforementioned idea that the cells can


Fig. 1. (a)-(c) The total strain response as a function of cycle number for the as-built, $900^{\circ} \mathrm{C}-10 \mathrm{~min}$ and $1050^{\circ} \mathrm{C}-10 \mathrm{~min}$ samples under $\Delta \sigma_{T}$ of 769 MPa , 762 MPa and 754 MPa , respectively. (d) Total strain range versus normalized number of cycles ( $\mathrm{N} / \mathrm{Nf}$ ) curves for the samples and fatigue conditions in Fig. 1(a)-(c). (e) The details of the curves within the normalized cycle number of 0.2 . (f) $92 \%$ fatigue life hysteresis loops for the as-built and $1050^{\circ} \mathrm{C}$ - 10 min samples tested at $\Delta \sigma_{T}$ of 769 MPa and 754 MPa , respectively. (g) Total stress range versus number of cycles to failure.


Fig. 2. Microstructures prior to deformations (a-f) and post-fatigued ( $\mathrm{g}-\mathrm{h}$ ) of the as-built sample ( $\mathrm{a}, \mathrm{c}, \mathrm{e}, \mathrm{g}$ ) and $1050^{\circ} \mathrm{C}-10 \mathrm{~min}$ sample (b, d, f, h), respectively. (a, b, g1, h1) Overlaid BC and orientation maps. Average grain size of each state was marked in the upper right corner. (c)-(d) and (e)-(f) SEM and bright-field TEM micrographs showing the typical configurations of dislocations before deformation. (g2) and (h2) KAM maps.


Fig. 3. Bright-field TEM micrographs of post-deformed as-built sample under different cyclic stresses revealing typical dislocation structures including (a)-(b) slip bands (SB). (c)-(d) and (f)-(g) Large population of stacking faults (SFs) origin from cell walls. (e) and (h) Some deformation twins present in the microstructures, and twintwin, twin-dislocation, and twin-cell structure interactions also occurred. (i) A HRTEM micrograph showing the atomic structures of the bunched nanotwins and twin boundaries marked by yellow lines.


Fig. 4. Bright-field TEM micrographs showing the representative post-deformed microstructure of the $1050^{\circ} \mathrm{C}$ - 10 min sample under different cyclic stresses. (a)-(b) Welldefined dislocation substructures, such as cells (Fig. 4a) and veins (Fig. 4b). (c), (d) and (e) Enlarged images of cell boundaries, consisting of dense dislocation networks. (f) A small amount of SFs formed in the deformed microstructure.
promote the formation of DTs. Fig. 3i shows a High-resolution TEM (HRTEM) micrograph of DTs; the thickness of them ranges from three atomic layers to about 10 nanometers, which supports that the growth mechanism of twins in our case appears to be layer-by-layer [1].

In deformed $1050^{\circ} \mathrm{C}-10 \mathrm{~min}$ sample, as present in Fig. 4, most dislocations arrange themselves from the random distribution state (Fig. 2f) into energetically favorable configurations, leading to the formation of well-defined wavy dislocation substructures, namely dense dislocation walls separated by channels with low dislocation density. The typical configurations of the substructures can be divided into two types: some of them are parallel to each other (Fig. 4b), called as vein-like structures; others tend to form irregular shapes (Fig. 4a), known as cell-like structures. From Fig. 4c-e, it can be seen that the dislocation networks (marked by yellow arrows) constitute the vein and/or cell-like substructures, whose
role is to maintain compatible deformation across different microstructures during the plastic deformation [27]. Additionally, unlike the as-built sample (Fig. 3), SFs only can be occasionally observed in the deformed $1050^{\circ} \mathrm{C}-10$ min sample, and no DTs are revealed. For 316LSS, the critical twinning stress was reported to be around 840 MPa by both experimental and theoretical approaches [28,29], which is indeed much higher than the cyclic stress amplitudes adopted in our study. This further confirms that the cell structures in AM 316LSS contribute to the formation of SFs and DTs.

The cyclic strain response of materials is highly determined by the evolution of microstructures. For heat-treated samples, the rearrangement of dislocations from a random distribution state into the low energy configurations (e.g., cell-/vein-like substructures) is a dislocation density net reduction process, which results in initial cyclic softening. However, the cell structures in as-built sam-
ple are pre-existing before deformation, and thus no initial cyclic softening occurs. Upon further cycling, dislocations multiply and propagate to maintain compatible deformation across microstructures, and their interaction leads to the hardening effect. For the as-built sample, the unique 3D interactions between dislocations, DTs, SFs and SBs provide additional contribution to the hardening effect, which is not readily available in conventional counterparts. Especially the formation of DTs promoted by cell structures should have significant influences on dislocation motion through the dynamic Hall-Petch effect [22]. Once the multiplication and annihilation of dislocations reach an equilibrium state, no significant variation of dislocation densities in the microstructures, resulting in the steady cyclic response until failure. Moreover, owing to the ultrafast cooling rate of AM process, the chemical heterogeneity is formed within the cell structures [1,2]. The dislocation motion can be impeded by the coherent internal stress due to the chemical misfit between the enriched regions (like cell walls) and depleted regions (like cell interiors) [30]. Additionally, the spatial variation of elastic modulus between the enriched regions and depleted regions also leads to strengthening [30]. Therefore, the compositional micro-segregation could also contribute to the evolution of the cyclic stress response. Note that the formation of DTs, the additional 3D interactions, and cellular compositional microsegregation might effectively inhibit crack initiation and propagation, and are also responsible for the notably lower cyclic softening rate of the as-built sample in Fig. 1d.

From the aforementioned discussion, the distinct deformation mechanisms of the as-built and $1050^{\circ} \mathrm{C}-10$ min samples provide an in-depth understanding of the difference in fatigue property. Evidently, the dominant dislocation configurations in $1050^{\circ} \mathrm{C}-10 \mathrm{~min}$ sample are the cell-/vein-like substructures. They are closely related to the development of extrusions and intrusions on sample surfaces, where fatigue cracks can easily nucleate [31,32]. Conversely, due to the existence of pre-existing cell structures, the planar dislocation structures, SFs and DTs, are more frequently observed in the as-built sample, which delays the strain localization and lead to its superior fatigue performance [33]. Note that, in addition to cell structures, other factors, such as crystallographic orientation, cycle number and applied stress range, also have been reported to affect dislocation substructures [13,33-36]. For example, study by Nellessen et al. [35] showed that increasing strain amplitude and cycle number would increase the dislocation density and promote the formation of vein-like substructures. Besides, the mechanisms that promote the formation of SFs and DTs by cell structures under cyclic loading are still unclear, in-situ fatigue experiments with the help of HRTEM are suggested for further study.

In conclusion, the AM 316LSS with different concentrations of cell structures show notably different cyclic strain responses. Compared with annealed samples, the fatigue process of the as-built sample only consists of the steady and overload stages, without the initial softening stage. Moreover, the as-built sample possesses higher cyclic strength, lower cyclic softening rate, and longer lifetime. For all samples, microscopic analyses show no difference in grain orientation, dimension and shape. However, the distinct microstructure evolution investigated by TEM sheds light on understanding of the fatigue behaviors. In deformed as-built sample, the dominate dislocation configurations are planar dislocation structures, SFs and DTs, promoted by cell structures, leading to uniform strain accumulation. On the contrary, in the deformed $1050^{\circ} \mathrm{C}$ 10 min sample, dislocations arrange themselves from a random distribution state into the well-defined wavy dislocations structures (e.g., cell-/vein-like substructures), causing strain localization. Therefore, the fatigue performance of AM 316LSS was significantly enhanced by the cell structures via changing dislocation slip modes.

## Declaration of Competing interest

The authors declare that they have no conflict of interests or personal relationships that could have appeared to influence the work reported in this paper.

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## Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.scriptamat.2021. 114190.

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