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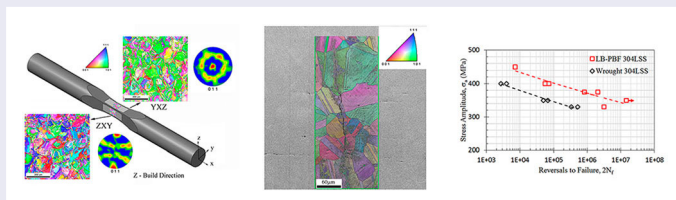
Additive manufacturing of fatigue resistant austenitic stainless steels by understanding process-structure–property relationships

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ABSTRACT

The limited understanding of additive manufacturing process-structure–property inter-relationships raises some concerns regarding the structural reliability, which limits the adoption of this emerging technology. In this study, laser beam-powder bed fusion is leveraged to fabricate an austenitic stainless steel with a microstructure containing minimal known crack initiation features. Ex-situ microstructural observations of the crack initiation features and mechanisms are carried out for interrupted fatigue tests via electron backscatter diffraction mapping of the micro-cracks. Results show that the additive manufactured stainless steel alloy has improved fatigue resistance compared to its wrought counterpart as a result of the unique microstructural features.



IMPACT STATEMENT

Using an experimental ex-situ microstructural investigation, the fatigue resistance of additive manufactured 304L stainless steel was shown to be superior to the wrought counterpart by avoiding the typical failure mechanisms.

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KEYWORDS

Laser beam-powder bed fusion; crack initiation; microstructure; twin boundary; cyclic deformation

Introduction

Additive manufacturing promises to revolutionize the fabrication process of structural components in several industries. The layer-by-layer fabrication process results in a wide design-space with the freedom to create complex geometries and, more importantly, the ability to have direct localized control of the thermal history. There is an intimate relationship between the thermal history and the resulting microstructure [1], such that tailored, or even functionally graded, microstructures may be realized through additive manufacturing [2]. For example, in areas where high strength and resistance to fatigue crack initiation (FCI) is required, high cooling rates may be beneficial to produce finer microstructures. On the other hand, as larger grains are beneficial for areas where crack

growth is critical, a reduction in the cooling rate can lead to a coarsening of the microstructure [3].

The extension of additive manufacturing to fatigue critical applications has been hindered by process-induced defects such as lack-of-fusion (LoF), gas entrapped pores, and surface roughness [4–6]. Defects act as stress concentrators inducing early FCI, severely limiting the potential for producing reliable, fatigue resistant additive manufactured (AM) parts. This study aims to better understand process-structure–property relationships as they relate to the fatigue performance of laser beam-powder bed fusion (LB-PBF) 304L stainless steel (304LSS). The majority of research efforts regarding AM parts have focused on understanding the structure–property relationships as they are related to

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processed induced defects [7–11]. This work instead focuses on materials that are less sensitive to defects to elicit the microstructure's role in FCI of AM materials. The results presented in this study give early insight into the true potential of additive manufacturing to produce targeted microstructure for application-specific desired properties.

Crack initiation in austenitic SSs

Austenitic stainless steels (SSs) in their wrought form are known to be more susceptible to FCI at annealing twin boundaries ($\Sigma 3$ -TB) and high angle grain boundaries (HAGB) than nano/micro-scale defects such as inter-metallic inclusions and voids [12–15]. The deformation behaviour at $\Sigma 3$ -TB and HAGB has been shown to result in both elastic and plastic incompatibilities across the boundary [13–20]. For low applied stresses, such as those in the high-cycle fatigue (HCF) regime, the small differences in elastic deformation near $\Sigma 3$ -TBs result in elastic mismatches across the boundaries. The increased localized stresses promote FCI along the boundary which can be exacerbated when higher loads are applied, expediting the crack formation. Additionally, HAGB are also capable of promoting FCI in wrought austenitic SSs as dislocations are not easily transferred across the grain boundary. As these dislocations pile up, edge dislocations of different signs attract each other resulting in the accumulation of vacancies and the formation of micro-cracks [21].

While microstructural features such as $\Sigma 3$ -TB/HAGB are difficult to avoid in the manufacturing of wrought materials, additive manufacturing provides a means to produce net shaped/near-net shaped parts without the need for post-processing steps such as forming and solution annealing. Austenitic SSs exhibit exceptional toughness and have been successfully implemented in LB-PBF processes [22,23].

Ex-situ experiments to observe FCI mechanisms

Rectangular LB-PBF specimen blanks with dimensions of 13mm-x-13mm-x-70 mm were fabricated horizontally on an EOS-M290 and stress relieved at 400°C for 1 hr. The process parameters followed manufacturer recommended parameters for 316L SS with a laser power of 195 W, scan speed of 1083 mm/s, and a hatch distance

of 90 μm . The powder feedstock was provided by LPW with a particle size distribution of 15–45 μm and composition given in Table 1. Specimens were then machined to a cylindrical uniform gage section following ASTM-E606 [24] and polished longitudinally to a mirror finish. Non-traditional square gage crack initiation (CIS) specimens were also machined and electro-polished, as previously described in [14], to investigate the FCI mechanisms associated with LB-PBF(304LSS).

All standard fatigue specimens were tested until failure using a servo-hydraulic test frame with a 100 kN capacity. Quasi-static tension tests were performed at a strain rate of 0.001 s^{-1} . The CIS were tested under force-control only and were interrupted periodically and investigated using a Zeiss FE-SEM with an EDAX-EBSD detector and OIM software. Micro-cracks were located and mapped using EBSD to observe the microstructural characteristics associated with FCI.

Establishing process-structure-property relationships

Differences in microstructure in relation to the drawing and build direction for wrought and LB-PBF(304LSS), respectively, are shown in Figure 1. The YXZ plane relates to the faces which were perpendicular to the build direction while the ZXY plane relates to the faces parallel to the build direction. The LB-PBF microstructure analysis revealed a strong $\langle 011 \rangle$ texture as a result of the $\langle 001 \rangle$ easy growth direction and the high solidification rate [25–27]. Additionally, the LB-PBF microstructure evolution results in a much finer average grain size (12 and 13 μm in the YXZ and ZXY planes, respectively) than the wrought counterpart (54 μm equiaxed). The LB-PBF material also showed a high density of low angle grain boundaries (LAGB) ($> 15^\circ$) and a low density of HAGB ($> 45^\circ$) and $\Sigma 3$ -TB ($\sim 60^\circ$), as shown by the misorientation histograms in Figure 1. This contrasts with the wrought material which shows a higher density of HAGB and $\Sigma 3$ -TB boundaries. The measured grain boundary lengths and number fractions for LAGB (5° – 15°), grain boundaries in the range of 15° – 45° , HAGB (45° – 54°), and $\Sigma 3$ -TB are given in Table 2.

The cyclic deformation behaviour of wrought 304LSS results in complex stress responses as affected by microstructural characteristics and evolution [28]. The cyclic deformation behaviour for the LB-PBF(304LSS)

Table 1. Chemical composition by weight % of the LB-PBF (AM) powder feedstock and wrought (WR) material provided by manufacturer.

	C	Cr	Cu	Mn	N	Ni	O	P	S	Si
AM	0.017	18.40	< 0.10	1.20	0.09	9.40	0.03	0.012	0.005	0.55
WR	0.029	18.06	0.45	1.35	0.07	8.59	NA	0.032	0.030	0.28

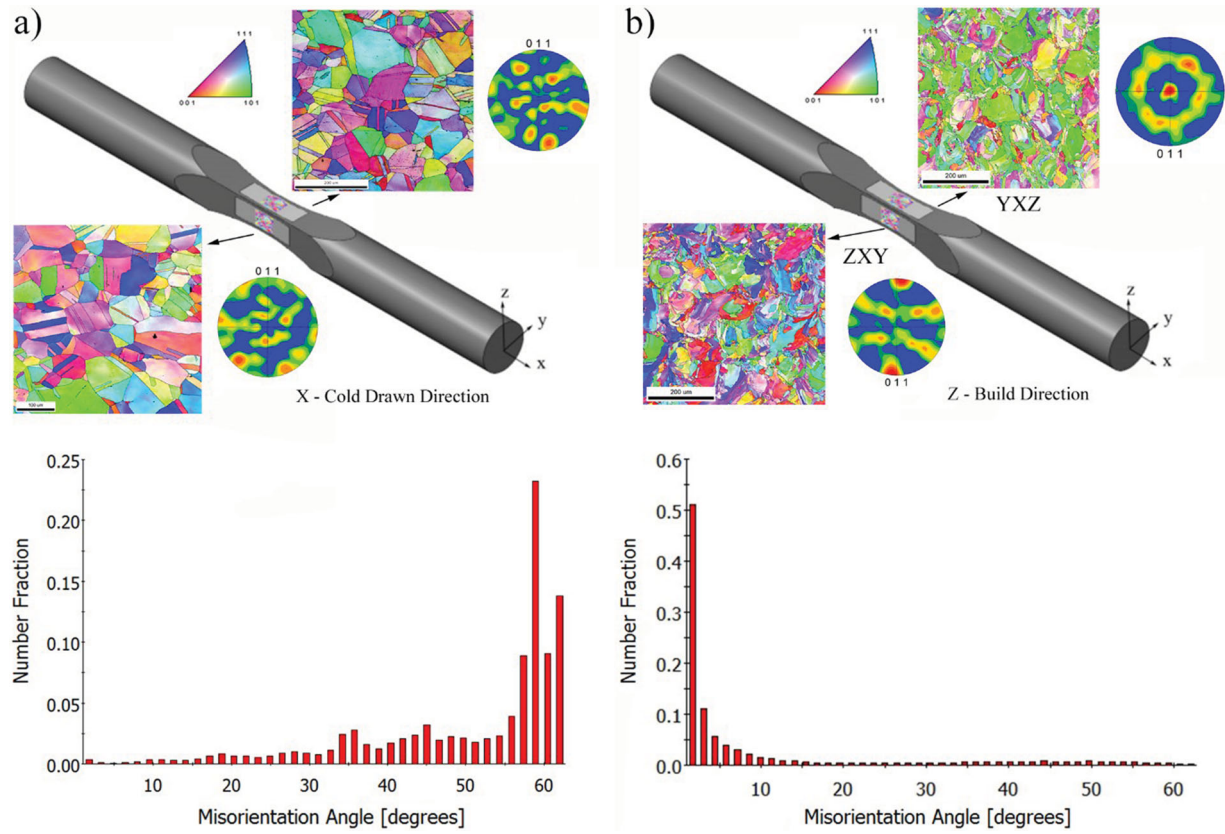


Figure 1. Microstructure; analysis for (a) cold drawn and annealed wrought 304LSS and (b) LB-PBF(304LSS) for each face in relation to the drawing or build directions, respectively.

Table 2. Grain boundary lengths and fractions for both wrought and LB-PBF 304LSS.

		5°–15°(LAGB)	15°–45°	45°–54°(HAGB)	$\sum 3\text{-TB} \pm 8.66$
LB-PBF	Length (mm)	43.8	33.5	14.4	1.51093
	Fraction	0.144	0.11	0.047	0.005
Wrought	Length (mm)	1.31	7.74	4.08	9.95063
	Fraction	0.014	0.082	0.043	0.105

under strain-controlled conditions, given in Figure 2, is similar to the observed behaviour for its wrought counterpart [28]. The stress response in Figure 2(a) reveals three distinct deformation stages: initial work hardening, cyclic softening[14], and secondary hardening [29]. The secondary hardening stage has been correlated to the deformation-induced martensitic phase transformation that occurs for 304LSS [28].

The complex stress response of 304LSS results in non-stabilized hysteresis loops during cyclic loading as the stress amplitude evolves through the related deformation stages. The half-life hysteresis loops are presented in Figure 2(b) along with the quasi-static tensile behaviour. The initial work hardening of the material at the strain amplitude of 0.2% results in a higher peak stress at half-life compared to the quasi-static tensile behaviour while the next strain amplitude step of 0.5% falls below the quasi-static stress-strain curve. Increasing the strain

amplitude results in further cyclic softening as observed for strain amplitudes of 0.75% and 1.0%.

Figure 2(c) shows the cyclic stress-plastic strain relationship for both wrought and LB-PBF(304LSS). The LB-PBF material shows a slightly lower cyclic hardening rate, as indicated by the lower slope of the curve, as well as a lower fatigue strength coefficient (σ_a at $\epsilon_{a-p} = 1.0\%$). However, the LB-PBF material exhibits a much higher cyclic yield strength ($\sigma_y' = 424$ MPa) than the wrought material ($\sigma_y' = 334$ MPa). This is mostly attributed to the finer microstructure and smaller slip length of the LB-PBF material.

The quasi-static tensile engineering stress-strain and fatigue behaviour of the LB-PBF material in comparison to its wrought counterpart are presented in Figure 3(a–c). The LB-PBF material shows a slightly higher tensile yield strength and elongation to failure with a slightly lower ultimate strength. The increased yield strength can

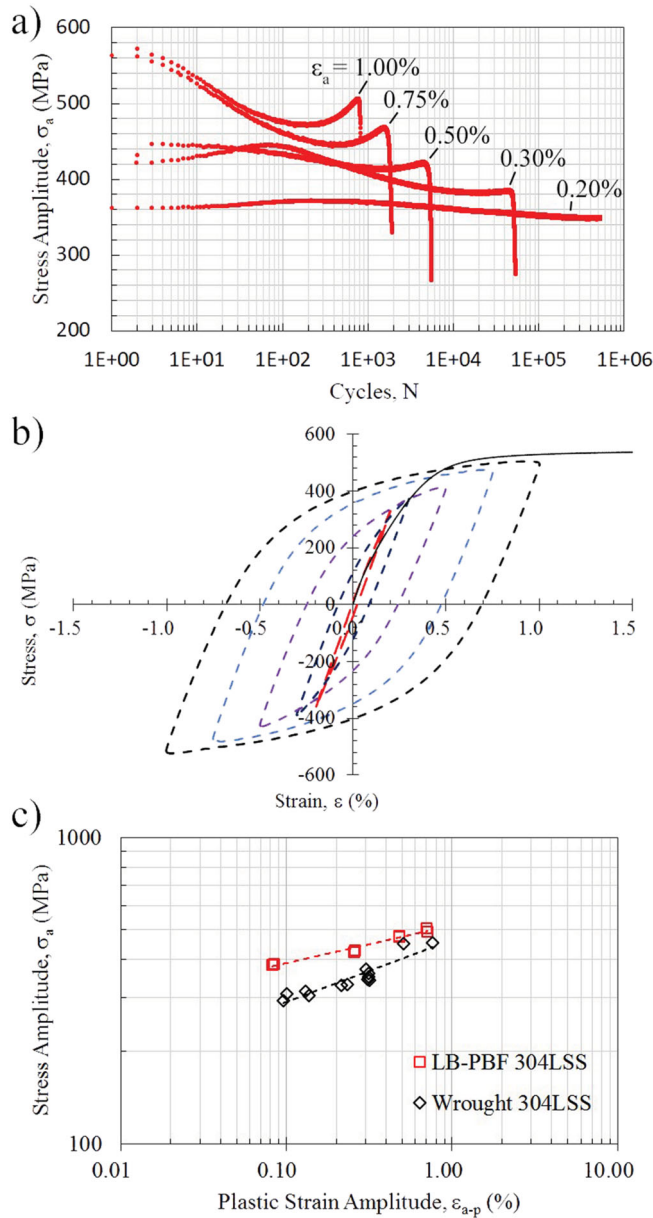


Figure 2. Cyclic deformation behaviour of LB-PBF showing (a) stress response under strain-controlled conditions, (b) cyclic stress-strain hysteresis loops, and (c) strain hardening behaviour in comparison with wrought material.

be related to the finer microstructure observed for the LB-PBF material. It should be mentioned, however, that the wrought material was evaluated using standard E8 test specimen geometry while the AM materials did not strictly follow the ASTM E8 [30] requirements.

The strain-life fatigue data and curves shown in Figure 3(b) indicate the LB-PBF material has slightly higher fatigue resistance in the low-cycle fatigue (LCF) regime, while the HCF behaviour is very similar to the wrought counterpart. The improved LCF resistance can be explained by the higher cyclic yield strength of the LB-PBF resulting in a reduced level of extrusions and

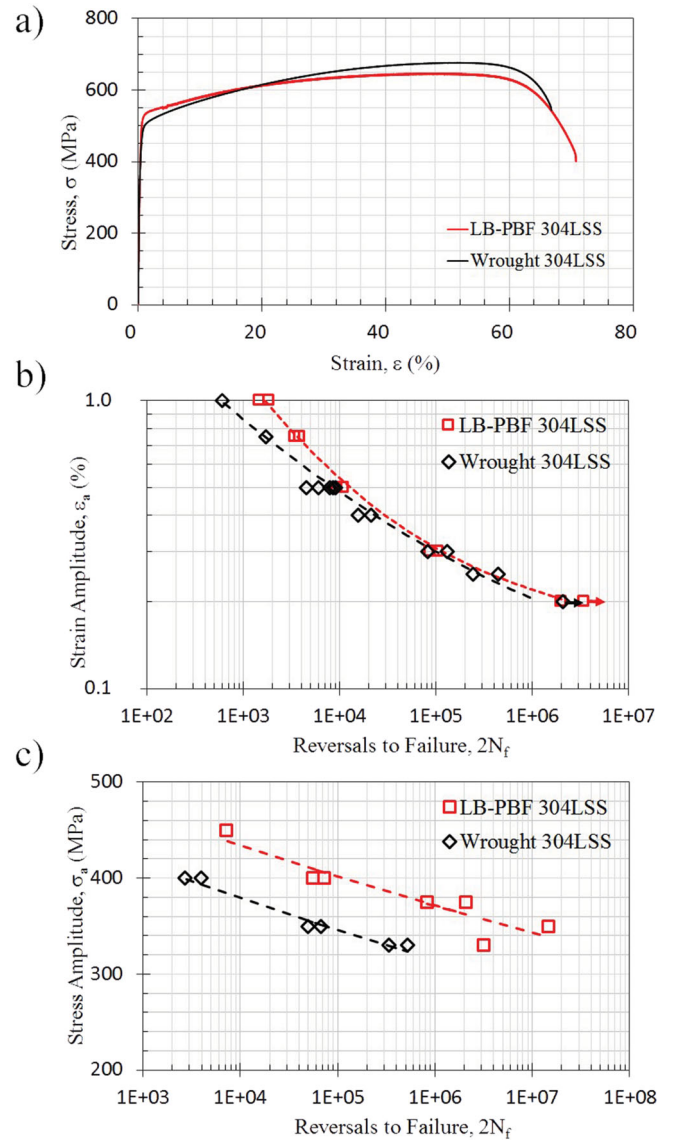


Figure 3. Comparison of LB-PBF and wrought 304LSS mechanical behaviour: (a) quasi-static tensile behaviour, (b) strain-life fatigue behaviour, and (c) stress-life fatigue behaviour.

intrusions on the surface. The similarity of the wrought and LB-PBF HCF fatigue behaviour suggests that defects such as LoF and gas entrapped pores, which typically deteriorate the HCF performance of most AM materials, are not greatly influencing the LB-PBF(304LSS).

The stress-life fatigue data and corresponding Basquin curves shown in Figure 3(c) remarkably indicates the LB-PBF(304LSS) has improved stress-life fatigue resistance across all stress levels. The fatigue life of LB-PBF material for any given stress amplitude was one-two orders of magnitude longer than the wrought material. While most AM materials fail under cyclic loading due to the presence of process-induced defects [6,9,31,32], the majority of the dominating cracks in the LB-PBF(304LSS) specimens initiate at the surface without the presence of

defects. Figure 4 details two fracture surfaces from (a) strain control test at $\epsilon_a = 0.3\%$ and (b) force control test at $\sigma_a = 400$ MPa. In both cases a dominant crack initiates at the surface (indicated by arrow-1) without any indication of the presence of defects then coalesces with a smaller crack which shows evidence of FCI at a defect (indicated by arrow-2). Surprisingly, in these cases and for many other specimens, the dominant crack did not initiate from a defect suggesting that microstructural features are responsible for crack initiation.

The dominating effects of microstructure rather than defects occur despite the defect sizes being much larger than the average microstructure size. Previous works have shown improved fatigue performance of AM titanium alloys after HIP related to defect sizes being smaller than the grains [33–35]. For the present study, however, no HIP was performed and defects present in the material were much larger than the grain size, as indicated by Figure 4.

Considering the majority of the fatigue life in HCF regime is spent in nucleating a crack, the observed differences in the 304LSS microstructural features, are most likely responsible for the improved HCF behaviour. Under strain-controlled conditions, the similar HCF behaviour between wrought and LB-PBF 304LSS can be explained by the deformation being limited by the utilized control mode. Contrastingly, under force-controlled conditions, the deformation for the LB-PBF material with a higher cyclic yield strength would be expected to be less severe than the wrought counterpart. The lower cyclic deformation leads to improved fatigue resistance for the LB-PBF(304LSS).

The ex-situ CIS results for both the wrought and LB-PBF material at the stress amplitude of 330 MPa are shown in Figure 5 with their corresponding inverse pole figure (IPF) orientation overlays. The crack for the wrought material (Figure 5(a)) developed into a dominant crack approximately 310 μm long by 35,000 cycles,

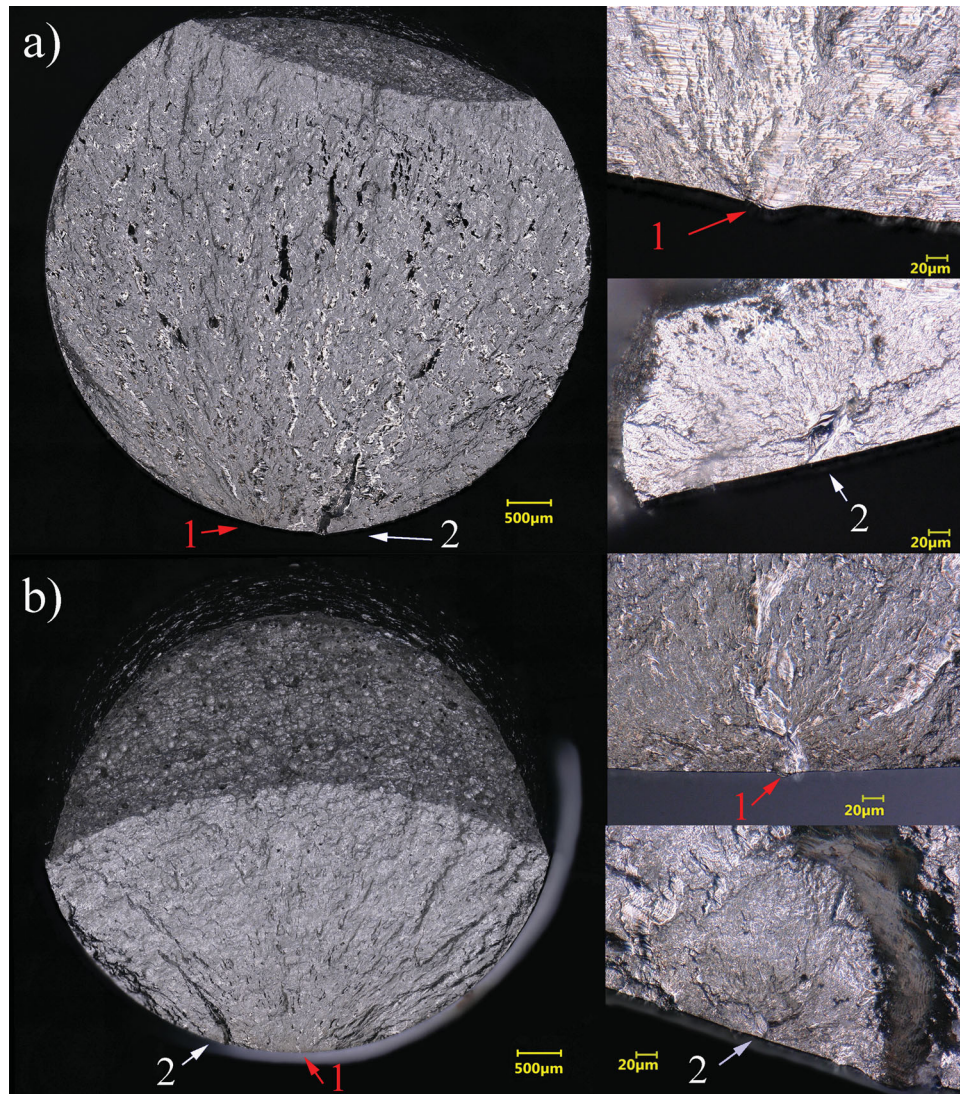


Figure 4. Fracture surfaces of (a) strain control test at $\epsilon_a = 0.3\%$ and (b) force control test at $\sigma_a = 400$ MPa.

while the longest crack observed in the LB-PBF material (Figure 5(c)) was still in the initiation stage at 76 μm long by 100,000 cycles. The overlays of the EBSD orientation data on the LB-PBF cracks in Figure 5 show the relationship between the crack and underlying microstructure. The crack in Figure 5(c) occurred at a HAGB approximately 51° and propagated along the boundary until it reached a LAGB. The crack shown in Figure 5(b) initiated at a $\Sigma 3$ -TB boundary which was surprising

considering the lack of these boundaries within the LB-PBF(304LSS) microstructure. Interestingly, the $\Sigma 3$ -TB is oriented approximately 45° from the loading direction similar to what was observed for the wrought material [14].

The microstructural CIS investigation revealed that the majority of cracks in the LB-PBF material initiated at HAGB. This observation is in contrast to the wrought material where FCI was dominated by $\Sigma 3$ -TB

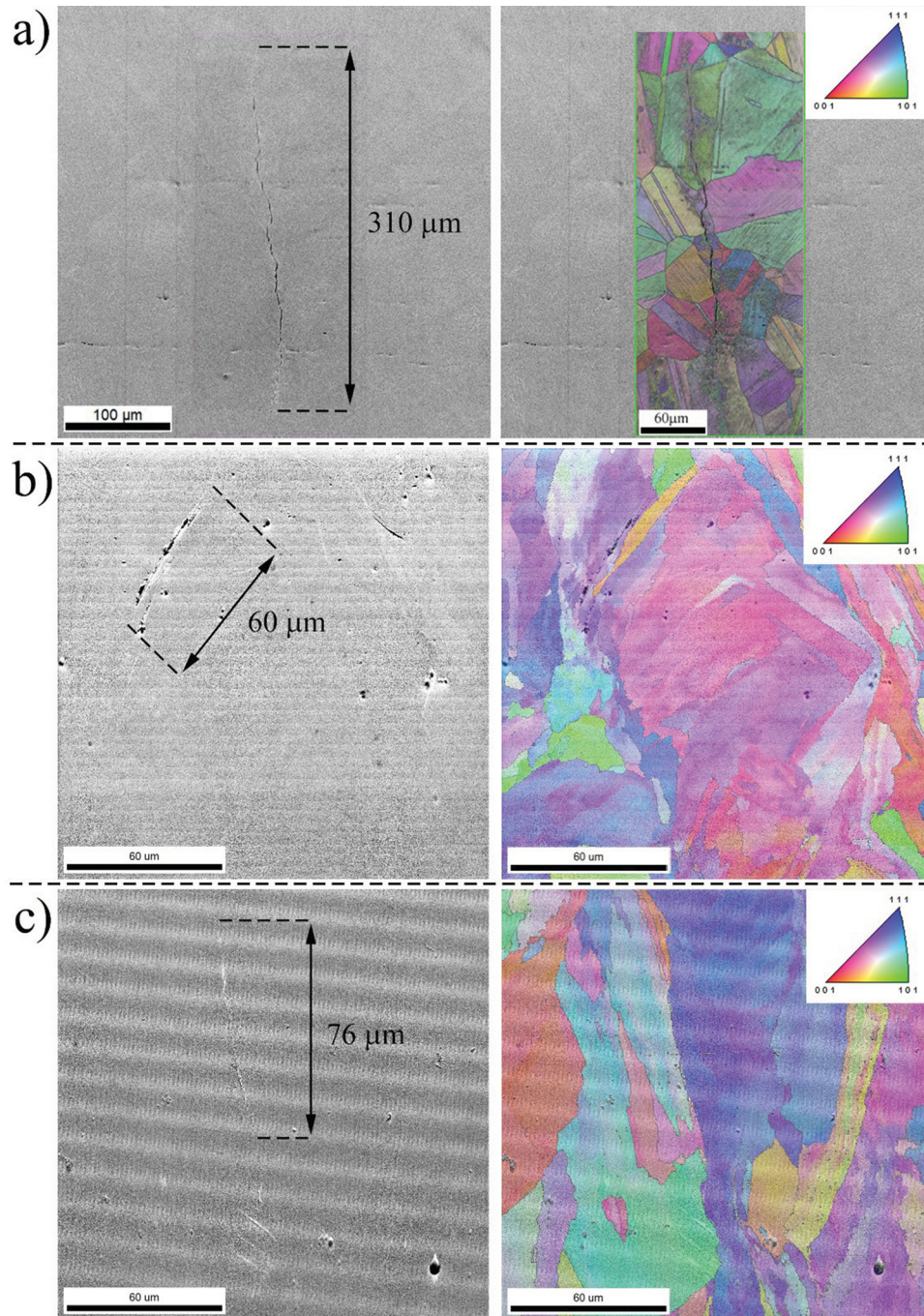


Figure 5. (a) SEM image of a dominant crack in the wrought material with the grain structure overlay, (b) SEM image of a $\Sigma 3$ -TB boundary crack in LB-PBF material with the grain structure overlay, and (c) SEM image of a HAGB crack in LB-PBF material with the grain structure overlay.

[14]. The solidification characteristics of LB-PBF process, and subsequently, the presence of $<011>$ texture results in a high density of LAGB and lower density of HAGB/ Σ 3-TB. The nature of FCI in 304LSS and the unique microstructure for LB-PBF 304LSS, lead to a reduction in Σ 3-TB, shifting the dominant FCI mechanism of the LB-PBF(304LSS) to HAGB. More importantly, the higher cyclic yield strength of LB-PBF(304LSS) results in much less cyclic deformation, even at stress amplitudes approaching the quasi-static yield stress. This is noticeable in Figure 4, where the slip lines, that are easily distinguishable in Figure 4(a) for the wrought material, are mostly absent in Figure 4(b,c) for the LB-PBF material. This combination of finer microstructure and lower HAGB/ Σ 3-TB density results in the improved fatigue performance for LB-PBF(304LSS) compared to its wrought counterpart.

Conclusions

These profound results show that while most AM materials are susceptible to early fatigue failures, austenitic SSs can be successfully fabricated via LB-PBF to offer superior strength, ductility, and fatigue resistance compared to their wrought counterparts. Taking advantage of the intimate relationships among the localized thermal input during the AM process and the resulting microstructure and mechanical properties, the fatigue performance was improved by avoiding the typical FCI mechanisms associated with the wrought counterpart.

These findings for LB-PBF(304LSS) offer early insight into how additive manufacturing can be used to fabricate superior fatigue resistant materials by further establishing the process-structure-property-performance relationships and taking advantage of the increased design-space. By leveraging the intimate relationship between local thermal input and the resulting microstructure/properties it may be possible to tailor microstructure to meet specific loading requirements. The ability to fabricate AM materials with increased resistance to crack initiation and/or growth can expedite the adoption of this technology for fatigue critical applications across many industrial sectors to deliver lighter, stronger, and safer products.

It is important to note that the internal porosity is also influenced by the thermal history and deviations outside of an optimal process window could result in defects dominating the fatigue behaviour despite the improved microstructure. More in-depth studies focusing on various process/post-process effects on the resulting microstructure, dislocation density, and deformation characteristics are needed to fully establish the process-structure-property relationships for LB-PBF(304LSS).

Disclosure statement

No potential conflict of interest was reported by the authors.

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