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Cascading of the as-built microstructure through heat treatment and its role on the tensile properties of laser powder bed fused Inconel 718

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Abstract

The microstructures and mechanical properties of the Ni based superalloy, Inconel 718 (IN718), which was additively manufactured using the laser beam powder bed fusion (LB-PBF) technique in the as-built (AB) and heat treated (HT) conditions were investigated, with emphasis on the microstructural evolution from the AB to HT conditions and, in turn, on the tensile properties. Optimized LB-PBF parameters in two different processing instruments led to distinct textures in the AB alloys, which, in turn, impart diverging combinations of strength-ductility properties. These variations are rationalized by recourse to an analysis of the effective mean free path for dislocations within the solidification cell structures. Amongst the five different heat treatment schedules examined, one set led to the retention of as-built grain morphology and texture with the formation of the δ phase that is oriented at either ±45° to build direction in the alloy with <100> texture or at 0/±90° in the alloy with <110> texture. The second set of heat treatments led to complete recrystallization, and hence loss of as-built microstructural signatures, with no δ phase. The critical role of δ phase in determining the grain growth kinetics and subsequently the anisotropy in mechanical properties of heat treated IN718 was elucidated. The absence of δ phase was found to enhance both strength and ductility, while its occurrence in the alloy with <110> texture along the build direction reduces ductility markedly. Implications of these results in terms of developing and designing the processing strategies of LB-PBF Inconel 718 with tailored microstructures and good mechanical properties are discussed.

Keywords: *Laser Beam Powder Bed Fusion; Inconel 718; Heat treatment; Crystallographic Texture; Mechanical properties.*

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1. Introduction

Additive manufacturing (AM) processes such as the laser beam powder bed fusion (LB-PBF) have extended manufacturing freedom for producing complex parts, which are otherwise difficult to fabricate using conventional processes [1,2]. Consequently, there is considerable interest in establishing the processing-structure-mechanical property relations in AM alloys, especially those fabricated using LB-PBF and other such techniques due to the unique microstructures and textures in them in the as-built (AB) condition. A number of alloy systems including stainless steels [3,4], maraging steels [5,6], Al alloys [7–9], Ti alloys [10–12], and high entropy alloys [13,14] were explored hitherto. It was demonstrated that the Ni based superalloys such as Inconel 718 (IN718), Inconel 625, and Rene 42 are highly amenable to LB-PBF [15–17][18]. It was shown that LB-PBF IN718 that was subjected standard post-build heat treatment exhibits better tensile properties and similar fatigue properties as the alloy manufactured using the conventional means [19–22].

In the solutionized and aged condition, γ'' (Ni_3(Nb, Mo) with $D0_{22}$ body centered tetragonal (BCT) crystal structure) and γ' (Ni_3(Al,Ti) with $L1_2$ cubic structure) precipitates are the primary strengthening sources in IN718. The heat treatment schedule employed determines their size and distribution and hence the mechanical properties. In this pursuit, a number of innovative heat treatment schedules are proposed and exceptional strength – ductility combinations were demonstrated for conventionally manufactured IN718. In the context of LB-PBF IN718, Deng et.al. followed a longer homogenization, solution anneal and two step aging treatment in order to relieve the residual stresses and stored dislocations, and in the process achieved isotropy in the mechanical performance [23]. For imparting a high damage tolerance, a heat treatment schedule that would retain the mesostructure inherent to the LB-PBF process may be desirable.

A key drawback common to many of the metal AM processes is with respect to the reproducibility. Often, batch-to-batch and equipment-to-equipment variations in the properties of parts produced, even when the processing methodology employed is the same, are noted. Such variability is currently a major bottleneck for the certification and widespread adaption of this new manufacturing technology. Consequently, it is important to understand as to how the microstructural features in the AB parts determine the finished product's mechanical performance. Towards this end, we critically examine the role of initial microstructures and textures of LB-PBF IN718 that was produced using two distinct combinations of processing parameters, which result in vastly different microstructures in the as-fabricated condition, and heat treated in five different ways on the tensile properties. The connections between the

microstructural variations, including those associated with the presence of the Laves phases, which is not always desirable, and those of the tensile properties are examined in detail. These results demonstrate the cascading of the initial microstructural signatures through the heat treatments and the critical role played by them on the tensile properties. Consequently, they highlight the importance of microstructural control of AB LB-PBF IN718 for better reliability and reproducibility of the parts produced.

2. Materials and experiments

2.1. Materials

Pre-alloyed IN718 powders (size range: 10-50 µm; D50: 32 – 37 µm) were used for fabricating 64 mm (height) coupons that are 20 by 10 mm^2 in cross-section using two different LB-PBF machines (both equipped with the Nd: YAG fiber lasers and Ar gas atmosphere): Concept Laser M2 and Trumpf Truprint-1000. Hereafter, IN718 produced using these two machines are referred as LP1 and LP2, respectively. The optimized process parameters utilized for them are listed in Table 1. Since the laser spot sizes for LP1 and LP2 are 130 and 30 µm, respectively, a significant difference in the volumetric energy densities (VED) (~88.7 and 62.4 J/mm^3, respectively) exists. In both the cases, the random island scan strategy was employed, as it is known to minimize the residual stresses [24]. The islands in each layer were offset by 1 mm (for LP1) and 0.2 mm (for LP2) in both the in-plane directions. The printed blocks were sectioned from the substrate plates using electro discharge machining (EDM).

Microstructural and tensile property evaluation of the LB-PBF IN718 was performed in the as-built (AB) and heat treated (HT) conditions. For the latter, five different schedules that are listed in Table 2 were employed. These were decided on the basis of the following considerations: (i) In the AB condition, the elements responsible for forming the strengthening precipitates (during aging) such as Nb and Mo segregate along the cellular boundaries to form the Laves phase [4]. Solutionizing treatment is necessary in order to dissolve them into the γ matrix. The extent of dissolution is determined by the exposure temperature and time. Keeping this in view, three different solutionizing temperatures, 960 (HT1), 1030 (HT2) and 1100 °C (HT4 and HT5), were selected to retain different extents of segregates and attain varying proportions of the precipitates in the final microstructure. (ii) A direct two step aging treatment (HT3), which allows the retention of the cellular microstructure and meso-structure associated with LB-PBF process, was also explored. (iii) Three different aging schedules were followed to examine the effect of precipitate size on the tensile properties. One of them is a standard two step treatment: ageing at 718 °C for 8 h followed by further ageing at 621 °C for 8 h. This

procedure (referred to as AMS5663) is widely used in conventional manufacturing of IN718 parts and known to result in γ″ with size < 25 nm. This was used in both HT1 and HT4 schedules. A coarser γ″ is expected to form after the two step aging of 760 °C for 10 h followed by ageing at 649 °C for 8 h. This method was used in the HT3 and HT5 schedules. In HT2, further coarsening of γ″ was attained by a single step ageing at 790 °C for 8 h, as it is known to enhance ductility and hence fracture toughness [25].

2.2. Experiments

The relative density of the printed coupons was measured using the Archimedes' principle with a weighing scale capable of measuring weight to an accuracy of 0.0001 g. Microstructural characterization using optical microscopy and scanning electron microscopy (SEM) was carried out on mirror polished specimens with sandpaper of grit sizes ranging from 80 to 5 μm, diamond suspension and colloidal silica of size 1 and 0.05 μm, respectively. To observe the melt pool mesostructure, the specimens were electro-etched for 20 s with a (70% methanol and 30% H_2SO_4) electrolyte at a current and voltage of 0.95 mA and 5 V, respectively. Further microstructural investigations were carried out using back scattered electron imaging (BSE) and electron back scattered diffraction (EBSD) modes, for which the specimens were electro-polished for 30 s at a current and voltage of 2.5 mA and 38 V, respectively. The same electrolyte was used for both electro-etching and polishing. EBSD was carried out with a step size of 2 μm. The compositional variation in the specimens were acquired through energy dispersive spectroscopy (EDS) over an area of 2 x 2.5 mm^2. The EDS data were obtained from 100 random points to evaluate the distribution of all the major alloying elements. The grain size measurements were carried out using a commercially available software following the procedures recommended in ASTM-E112 [26]. To characterize sub-micron and nanoscale microstructural features, transmission electron microscopy (TEM) was conducted with a microscope operating at an acceleration voltage of 200 kV. The specimens for TEM were prepared by thinning down 3 mm diameter disc samples to < 100 μm thickness followed by dimple grinding to achieve ~20 μm thickness at the center of the discs. Low kV ion-polishing at a voltage of ~3 kV was conducted to acquire electron transparent regions. The microstructural features such as solidification cells, precipitate size and volume fraction were measured from SEM/TEM micrographs using ImageJ, a publicly available image analysis software.

For the uniaxial tensile tests, flat dog bone shaped specimens were machined with a gauge length, width, and thickness of 6, 2 and 1 mm, respectively. Tests were carried out at a

nominal strain rate of 10^{-3} s^{-1}, where the strain was measured using a video extensometer attached to the testing machine. All the tensile tests were performed according to ASTM standard: E8/E8M-11 [27]. Specimens were tested in two orientations such that the loading axis is either parallel or perpendicular to the build direction. Hereafter, these are referred as 'BD' and 'SD', respectively.

3. Results

3.1. Microstructures in the AB condition

The melt pool meso-structures of LP1 and LP2 are displayed in Figs. 1a and b, respectively. The melt pool width and depth in LP1 are 125 ± 10 and 46 ± 6 µm, respectively. In LP2, they are 66 ± 4 and 101 ± 10 µm, respectively. Note that the melt pool depth in LP2 is more than double that in LP1, while the width is nearly-half. These geometries of the melt pools indicate that the melting mode changed from the conduction mode in LP1 to the keyhole mode in LP2. Such a transition is usually associated with an increasing VED [28]. In the present case, however, LP2 has 30% lower VED compared to LP1. A modified Rosenthal equation, which is commonly used for predicting the geometry of a melt pool for the given combination of process parameters, suggests that this anomaly is related to significant difference in the laser spot sizes used for LP1 and LP2 [29]. The difference in the laser spot size and VED also results in different thermal gradients inside the melt-pool. The effect of them on the growth of solidification cellular structures inside the melt-pool can be gauged from the micrographs displayed in Figs. 1c and d for LP1 and LP2, respectively. The dendritic cells grow epitaxially in the direction parallel to the build direction and through the melt pools of several layers in case of LP1. Such epitaxial growth during solidification in LB-PBF process is the reason behind columnar grain growth that is commonly observed in many LB-PBF alloys [30]. In LP2, in contrast, they grow towards the center of the melt pool, resulting in an angle of ~45° between their orientation and the build direction. Since the cells grow towards the center of the melt pool from the opposite faces of the boundary, their growth is restricted to the same melt pool, i.e., cells do not grow across the melt-pool boundary of the same layer. Additionally, the 90° scan rotation in LP2 restricts epitaxial grain growth between one deposited layer to the subsequent layer. In contrast, cells grow in LP1.

The inverse pole figure (IPF) and pole figure maps obtained using EBSD and displayed in Figs. 2a and c indicate a strong <100> texture in case of the LP1 that is usually observed in case of metals with cubic crystal structure produced by AM processes [31]. On the other hand, the observed texture in the case of LP2 is weak (see Figs. 2b and d) due to restricted

growth of the cellular structure. From Fig. 2d, it is also observed that the dominant texture in LP2 is <110> due to the 45° tilt of the cells with respect to BD. Furthermore, the IPF Map of LP1 displayed in Fig. S1a shows <100> texture regardless of BD or SD orientation. Even though the overall texture in LP2 is weak, in contrast, the IPF map shown in Fig. S1b indicates different textures along BD and SD. The analysis of the EBSD maps from 8 different areas reveal the average grain size (misorientations >15°) to be finer in case of LP2 (~12 ± 10 μm as compared to LP1 (~18 ± 13 μm). The average low angle grain boundary fractions (LAGBs) with less 15° misorientation are ~42% and ~33% in case of LP1 and LP2, respectively. In addition, the sub-grains are ~6 – 8 times larger in size in LP1 as compared to LP2. The sizes of the solidification cells (λ), quantified using high magnification BSE images such as those illustrated in Figs.3a and b, are ~0.70 ± 0.16 μm and ~0.4 ± 0.07 μm in LP1 and LP2, respectively.

The compositions, evaluated through EDS point analysis, are listed in Table 3. As seen, LP2 has slightly higher Nb and Mo. This is related to relatively higher solidification rate in LP2, which is a consequence of the faster scan speed and a lower VED overall. At a higher scan speed, the tendency for solute trapping is higher if the solid interface velocity approaches the diffusivity of Nb and Mo elements [32,33].

The TEM bright field images displayed in Figs. 3c, and d indicate that a relatively higher fraction of Nb segregates to the cell boundaries in LP1 as compared to LP2. The sizes of the Laves phases that are dispersed inside the cellular boundaries range between ~163 ± 73 nm in LP1 and ~110 ± 55 nm in case of LP2. This correlates with the higher amount of Nb and Mo in the matrix in case of LP2 since the Laves phases consist of these elements. LP2 exhibits a higher dislocation density due to higher cooling rate experienced by it during processing. The relative porosities of LP1 and LP2 are 0.42 ± 0.07% and 0.69 ± 0.08% in specimens, respectively.

3.2. Microstructure evolution after the heat treatment

Two distinct microstructures, which depend on the temperature at which the solutionizing treatment (ST) was performed, were observed. For ST below 1100 °C, recrystallisation does not take place. Consequently, different grain morphologies, texture, and precipitates were observed in samples solutionized below and above 1100 °C. In the first set, which comprises of HT1, HT2 and HT3, the as fabricated grain morphologies were retained after HT with varying degrees of grain growth depending on the exposure temperature and time. This category also led to precipitation of orthorhombic δ (Ni_3Nb) along with the primary

strengthening phases, γ′ and γ″. The second set, consisting of HT4 and HT5, results in a substantial alteration of the grain morphology, with a concomitant loss of texture and a dominant γ′ and γ″ based microstructure with no δ precipitation. Hereafter, these sets are referred to as A and B, respectively.

Representative IPF maps obtained on the LP1 specimens after Set A (HT2) and Set B (HT4) HTs are shown in Figs. 4a and c, respectively. The <100> texture that was seen in the AB alloys remains unaltered after all the heat treatments. Grain growth was notable in Fig. 4a with average size increasing to ~74 ± 53 μm after HT2. Similar trend in grain size is also noted for other HT samples of Set A (see data in Table 4). In comparison, the grain size increments due to Set A HTs on LP2 is relatively lower.

Set B HTs led to microstructures with bimodal grain size distribution as shown by a representative IPF map of HT4 displayed in Fig. 4c. A closer examination of this image reveals two distinct features: (i) persistence of the columnar grains with strong <100> texture, and (ii) recrystallization followed by growth of equiaxed grains with no preferred orientation. The latter feature is related to the abnormal sub-grain growth observed first by Humphrey et al. [34] in {110}<001> Al crystals and Al – 0.05% Si alloy and then later by several others in Ni based superalloys [35–39].

In case of LP2, the as fabricated <110> texture in the BD orientation was preserved after all the different HTs, as illustrated in Figs. 4b and d for HT2 and HT4, respectively. In the SD orientation, in contrast, a reduction in the intensity of the texture after Set A HTs and elimination of texture altogether after Set B HTs was noted, as shown in Figs. S2c and d of SI, respectively.

As mentioned before, all the microstructures of both the alloys after Set A HTs consist of δ precipitates. In case of HT1 (ST ~960 °C), needle shaped δ precipitates were observed in both LP1 and LP2 specimens as shown in Figs. 5a and b, respectively. These precipitates are much coarser at the grain boundaries. Their size and morphology within the grains appears to vary from fine to coarse, as highlighted with boxed regions in Figs. 5a and b. The area fraction (A_f) of them was measured at three different locations and found to be ~10% for LP2 as compared to ~8.55% for LP1. The higher amount of Nb in solid solution and the larger stored strain energy in the form of dislocation networks in LP2 are the likely reasons behind the relatively higher A_f observed in it. The aspect ratios, Λ (= l/w), of the δ precipitates were found to be ~12 and ~19 in LP1 and LP2, respectively from the image analyses performed on 5 micrographs each. After HT1, the Laves phases, which were present in the AB condition, could not be detected, indicating that HT1 leads to the dissolution of them. The major diameter ($2R$),

V_f, and aspect ratio (Λ) of γ″ phase, measured by analyzing the TEM micrographs, are tabulated in Table S1 of SI. Precipitation of δ during solutionizing consumes Nb in a significant amount, which, in turn, leads to a finer γ″ of ~19 nm in both LP1 and LP2.

During HT2, with the ST temperature of ~1030 °C that is not favorable for the formation of δ [40], δ would only nucleate during the ageing treatment. By using the material modeling software JMatPro [41], we ascertained that the nucleation of δ phase only starts after ~5 hours of ageing at 790 °C (HT2). This provides enough time for significant grain growth, adding to what was already achieved after the solutionizing step as illustrated in Fig.4a. In addition, HT2 led to the dissolution of most of the cellular structure and the associated Nb rich zones as well as the Laves phases. Any remaining cells and Laves phases act as sites for the nucleation of coarse δ precipitates and their subsequent growth during the aging. Figs. 5c and d show precipitation of δ at the grain boundaries and prior cell boundaries (blue arrow), albeit in much lesser proportion as compared to the HT1 case. V_f of δ was measured to be ~3.5% and ~4.5% in LP1 and LP2 alloys, respectively. Coarse γ″ (>80 nm) homogeneously precipitated in both the alloys, as observed in the inset micrographs of Fig. 5c, and d marked by the yellow arrows. TEM analyses show the sizes of γ″ to be ~81 ± 17 and ~85 ± 13 nm in LP1 and LP2, respectively, (Table S1 and Figure S3b). The red arrows indicate the overgrown oxides formed during the LB-PBF process which were also reported by other researchers [42].

Microstructures of LP1 and LP2 after HT3 show δ precipitation at both the cellular and grain boundaries, as seen in Figs. 5e and f, respectively. Though direct ageing is not favorable for δ precipitation, ageing at 760 °C for ~8 h (according to JMatPro) is sufficient for the nucleation of δ. Further, due to high strain energy stored in the form of dislocation networks in the AB specimens, the nucleation of δ might have started earlier (than 8 h of ageing time). However, the grain size is much larger compared to HT1. This is because grain boundary pinning by δ (that form relatively late during ageing) that deters the grain growth in IN718 is not effective in HT3 as compared to HT1 [43]. An irregular to globular morphological transition of the Laves phase, which was accompanied with a reduction in size, was also noted. This suggests that the Laves phases dissolved in order to facilitate the growth of the δ phase, which is similar to that reported by Liu *et al* [44]. The LP2 specimens were observed to have relatively smaller Laves phase (~50 ± 20 nm) as compared to LP1 (~70 ± 22 nm). Also, a denser network of γ″, which nucleate at the cellular boundaries (as indicated by the arrows in the inset of Fig. 5f) with a gradually diminishing population towards the center of cells is noted. This is because LP2 has relatively narrower segregated regions. TEM analysis shows a bimodal distribution of γ″ in both the LP1 and LP2 specimens after HT3.

The high temperature of 1100 °C used for ST in Set B HTs (HT4 and HT5) induces bimodal and equiaxed recrystallized grains in the LP1 and LP2 alloys, as illustrated in Fig.4c and d, respectively. Precipitation of the carbides (MC and/or $M_{23}C_6$) along the grain boundaries was also observed (Figs. 6a and b). These carbides inhibit grain growth [45,46], resulting in relatively smaller grains as compared to those that underwent HT2 and HT3. TEM dark field micrographs displayed in Figs. 6c, and d show the precipitation of spherical and ellipsoidal precipitates after HT4 in LP1 and LP2 specimens, respectively. The SAED patterns obtained along the <100> zone axis (see Fig. 6e) confirmed these to be γ′ and γ″, respectively. While V_f of γ″ were estimated to be ~18 ± 1.5% and ~18.5 ± 1.2% in LP1 and LP2, respectively, the sizes of γ″ along its major axis were found to be ~26 ± 5 and ~26 ± 4 nm. Two step aging during HT5 resulted in a much coarser γ″ precipitates, as seen in Figs. 6f and g, with average sizes of ~60 ± 9 nm and ~60 ± 10.5 nm for LP1 and LP2 specimens, respectively. In turn, it resulted in lower V_f of ~11 ± 1.2 % in both the alloys.

In summary, HT1 to HT3 led to the precipitation of δ precipitates at grain boundaries and cell boundaries along with the heterogenous distribution of γ′ and γ″, which are the primary strengthening precipitates of IN718. Precipitation of grain boundary δ inhibited excessive grain growth by Zener pinning mechanism during HT1 [39,47]. Delayed precipitation of δ during HT2 and HT3 resulted in relatively coarser grains. HT4 and HT5 brought out homogenous precipitation of γ′ and γ″, with coarse MC dispersed at the grain boundaries. Lower fraction of LAGBs in the AB LP2 resulted in equiaxed grain structure after HT4 and HT5, as opposed to the bimodal grain structure observed in LP1 due to selective recrystallisation in some regions and abnormal grain growth in others.

3.3. Tensile properties in the as-built condition

Representative engineering stress vs. strain responses of LP1 and LP2 specimens tested in the build and scan orientations are displayed in Fig. 7a, while the average values of 0.2% yield strength (σ_y), σ_u, and elongation to failure (e_f) are listed in Table 5. In the AB condition, σ_y of LP2 alloy are ~26% and 23% higher in the build and scan orientations, respectively, than those of LP1 specimens. While σ_u are also higher (~12 and 14%, respectively), the high strength comes at the cost of ductility as e_f are lower (by 36% and 5%, respectively). The anisotropy in σ_y of LP1 and LP2 specimens is similar (17 and 14%, respectively), which reduces to ~6 and 8% for σ_u.

A comparison of the σ_y and σ_u values of the LP1 and LP2 of the present study with those reported in the literature for LB-PBF IN718 is made in Table S2. While σ_y and σ_u for LP1

specimens are similar to those of reported by Chlebus *et al.* [48] and Deng *et al* [23], those of LP2 specimens are similar to the ones reported by Wang *et al.* [49] and Huang *et al.* [50]. Further, the overall ductility of both LP1 and LP2 alloys is comparable to several published research works. A marginally lower e_f in LP2 might be because of relatively finer grains. Figs. 7b and c show the representative fractograph of failed LP1 and LP2 specimens, respectively. High magnification fractographs show typical dimple features associated with ductile behavior, where dimples are larger in LP1 specimens compared to LP2 reflecting the difference in e_f.

3.4. Tensile properties after the heat-treatment

Representative tensile engineering stress vs. strain responses of the heat-treated specimens are displayed in Fig. S4 of SI, and the σ_y, σ_u, and e_f values are listed in Table 5. The enhancement in properties with respect to their values in the AB condition is also listed. The following are some key observations. (i) The direct ageing treatment HT3 results in the highest strength (σ_y and σ_u) in both LP1 and LP2 alloys, which are similar to the values reported in literature [50,51]. (ii) Lowest increment in the strength was observed after HT2 treatment due to over aging of primary strengthening precipitates. (iii) In the build orientation, HT1, HT3, HT4, and HT5 enhanced σ_y by ~80 – 85% in LP1 and by ~60 – 65% in LP2. In the scan orientation, the corresponding enhancements are ~65% and ~45%, respectively. Slight variations in σ_y amongst different HTs exist, which will be discussed later in detail. (iv) While e_f of LP1 in the build orientation does not change after HT2, e_f consistently dropped by ~19% after all the other heat treatment schedules. A precipitous reduction in e_f of LP2 (by ~60%) after HT1, HT2 and HT3 occurs, while HT4 and HT5 lead to only a marginal enhancement (by ~2.5%). (v) In the scan orientation, e_f of both LP1 and LP2 dropped nearly equally after the Set A heat-treatments (HT1, HT2, and HT3). However, e_f of LP2 specimens increased by 15% after HT4. (vi) After the Set B treatments, σ_y and σ_u of LP1 remain similar, while LP2 show slightly higher σ_y and σ_u after HT5 as compared to HT4. In summary, Sets A and B HTs lead to distinct tensile behavior, which is possibly related to the differential evolution of precipitates in them, which is discussed in detail in next section.

4. Discussion

4.1. Tensile properties in as fabricated condition

A significant enhancement in σ_y of a variety of alloys fabricated using the LB-PBF process was observed in literature [23,48–50,52–57]. In most cases, this was attributed to the substantial reduction in the characteristic microstructural length scale due to the presence of the fine

solidification cellular structures in them [58,59]. Hence, the σ_y enhancement is reasonably well-rationalized by replacing the grain size in the Hall-Petch relation with the cell size. However, it has been suggested in a few studies that the observed strengthening is due to forest dislocations that are present along the cellular boundaries [23,60]. For example, Deng *et al.* [23] estimated the residual stresses in the build and scan orientation of IN718 based on the geometrically necessary dislocation (GNDs) and correlated it with the variation in the measured σ_y. Likewise, Gallmeyer et. al. demonstrated good correlation between σ_y and density of dislocations using the Taylor strengthening model [60]. Yet another suggested strengthening mechanism is dependent on the constituents of the segregated region at the cell boundaries. For example, Hadadzadeh et al. suggest that the strengthening behavior in AlSi10Mg alloy is due to the combination of cell walls, Si precipitates and dislocation hardening [61,62]. Recently, Zhang et.al. showed that the higher strength found in LB-PBF IN718 is due to a combined contribution of statistically stored dislocation and Orowan strengthening caused by the Laves phases [57]. In contrast, Gokcekaya et al. showed a correlation between the crystallographic orientation to the yield strength and suggested a diminishing Hall-Petch strengthening effect due to texture [63]. While it is still a matter of debate as to which is the predominant factor that contributes to the strength, all the suggested mechanisms are related to the cell size and orientation.

Keeping the above in view, we consider the cell boundaries as the sole obstacles for dislocation motion in the present work. The following are the two reasons behind this approach. (1) The density of dislocations is challenging to measure; the density of GNDs measured using EBSD is sensitive to the step and kernel sizes employed [64]. While an accurate assessment of the dislocation density can be made by deconvoluting the X ray diffraction peaks to remove the effect of crystallite size and defects, it is quite complex and is not pursued in the present work. (2) The segregated phases are irregular in shape and vary dramatically in size and homogeneity. Therefore, an accurate assessment of the strengthening caused by them using the Orowan loop mechanism around the Laves phases is difficult.

While the cells in both LP1 and LP2 are columnar, their growth directions differ because of effective thermal gradient, as seen in Figs.1c and d. This alters the geometric arrangement of the cells with respect to build direction of the specimens, as illustrated schematically for the BD and SD orientations in Figs. 8a to c. This results in a tensile loading orientation dependence of the mean free path (ψ) that dislocations traverse before encountering a cell boundary. In general, ψ is set to be $d/2$ (where d is the average grain diameter) for estimating the back stress generated by the dislocation pile ups in conventionally manufactured

alloys, with an implicit assumption that dislocations nucleate at the center of the grain [65]. However, the probability dislocation nucleation at the cell boundaries is here in the current context due to segregation [66]. Fig. 8a illustrates the geometry when loading is parallel to <100>, which is representative of LP1 and LP2 tested in SD. If a dislocation nucleates near the cell wall, say at position I, it will advance along the maximum shear stress direction, which coincides with <110> due to the strong texture (as shown by the red arrow) at ~45° to the loading direction, with $\psi = \lambda$ due to its symmetrical shape. For tensile loading parallel to BD in LP1, which has <100> texture, the loading axis makes ~45° with <110> along which the dislocations propagate, as illustrated in Fig. 8b. Due to the columnar nature of the cells, $\psi = \sqrt{2}\lambda$. The situation is similar in case of LP2, as shown in Fig. 8c, except that the CRSS contribution will differ by the Taylor factor, *M*. Tensile properties of the as-built LB-PBF IN718 (measured in both BD and SD) from several published works along with the ψ estimated from their microstructures are tabulated in Table S2 of SI.

To examine the above hypothesis about ψ as the characteristic length scale for strengthening (instead of λ), σ_y is plotted in Fig. 8d as a function of $\psi^{-0.5}$ following the Hall-Petch relationship: $\sigma_y = \sigma_{fr} + K_y (\psi)^{-0.5}$, where σ_{fr} is the lattice friction stress, K_y is a locking parameter. Note this approach is necessary to address the anisotropy in the strength of AM alloys, which is often ignored even though columnar shape of the cells is commonly observed and reported. A linear fit through the σ_y - $(\psi)^{-0.5}$ data gives σ_{fr} of ~361 MPa and K_y ~334 MPa$(\mu m)^{0.5}$.

An analytical value of σ_{fr} can be obtained from the equation: $\sigma_{fr} = M\tau_{CRSS} + \sigma_{SSS}$, where τ_{CRSS} = 17.5 MPa for IN718 [67,68], *M* are ~ 2.6 and 3.1 (obtained from EBSD) for LP1 and LP2 specimens along BD, respectively, σ_{SSS} is the strengthening arising from the solid solution. The latter was estimated to be (~320 MPa) using the equations provided by Gypen [69] from the compositions of LP1 and LP2 specimens listed in Table 2. A good agreement between the estimated value of σ_{fr} (~370 MPa) on this basis and the fitted σ_{fr} value (361 MPa) supports the notion of ψ and its effect on the mechanical anisotropy in LB-PBF alloys with columnar cells.

Turning to ductility, there are several possible mechanisms that control it. For the alloys with grain size larger than a micron, coarsening of the grains often leads to enhanced ductility [70,71], while some reported the an opposing trend [72]. Ni *et al.* associated the anisotropy in ductility to the alignment of columnar grains with the tensile axis in LB-PBF IN718, as such grains are less prone to Mode I cracking and delay tensile failure [73]. In other

works, reduction in ductility is attributed to cracking of the segregated Laves phase and matrix during large deformation [51]. e_f of LP2 in the BD orientation is significantly smaller than that of LP1. Thus, the observed higher ductility in LP1's can be attributed to the coarser columnar grain oriented along the loading direction, as confirmed by the distribution of grains aligned to SD in Fig. S5. [73]. Likewise, the orientation dependence of e_f in LP1 can also be rationalized by the higher fraction of grain boundaries perpendicular to loading direction. On the other hand, Fig. S5 shows that there is no preferred orientation of grains in LP2 with respect to loading axis as seen in LP1. Yet, e_f of LP2 in BD is ~19% lower compared to SD, even though the effective grain size in BD is 15% higher. This is possibly due to the difference in the textures of LP1 and LP2. Although polycrystalline, both are highly textured. For instance, strong <100> texture is observed in LP1 as compared to slightly weaker <110> texture in case of LP2 (Figs. 2c and d). For example, predominance of slip planarity for loading parallel to <110> leads to a relatively lower strain hardening exponent, n, in the early stages of plastic deformation (n ~0.13). Loading along <100>, on the other hand, leads to cross-slip right after macroscopic yielding, resulting in higher n (~ 0.19) [74]. A relatively low n in LP2 specimens can lead to an earlier onset of necking resulting in lower e_f.

4.2. Yield strength of the heat treated alloys

The grain morphologies and textures of the as-built alloys were retained after heat treatments to varying proportions depending on the solutionizing temperatures. This has also led to diverse size distribution and V_f of strengthening precipitates, which, in turn, influence the tensile properties. Since the microstructures of LP1 and LP2 in the AB condition are distinct, the impact of various heat-treatments on the microstructure evolution is also different. In this section, the effect of such microstructural evolution due to HT on the yield strength is discussed, with emphasis on understanding the relative variation of it within the different heat treated alloys.

Even though the solutionizing temperature in set B heat-treatments is 1100 °C, LP1 specimens have bimodal grain structure due to partial recrystallisation, while LP2 specimens consist fully recrystallized, equiaxed grains (see Figs. 4c and d). Nevertheless, both the specimens retain their initial individual texture to some extent. The average aspect ratios of the grains (Λ_d) of LP1 and LP2 specimens after heat treatments are listed in Table 4. A higher Λ_d in LP1 indicates a larger proportion of columnar grains compared to LP2. The set B heat-treatments resulted in complete elimination of the cell structures while the set A treatments result in the cells decorated by δ precipitates. Therefore, one can anticipate that σ_y of the heat-

treated specimens would be affected by the grain size/morphology instead of cell size/morphology.

To investigate the impact of the morphology of the grains on the anisotropy in σ_y, the difference between σ_y of SD and BD orientations, $\Delta\sigma_y$, is plotted in Fig. 9a as a function of Λ_d. As seen, $\Delta\sigma_y$ increases sharply between Λ_d values of 2 and 6 and remains invariant at ~110 MPa for $\Lambda_d > 6$. LP1 specimens, which have relatively higher Λ_d compared to LP2, exhibit a larger σ_y differential. The observed influence of grain morphology can be rationalized through a mechanism that is analogous to the one used for the cell morphology on σ_y of the alloys in the AB condition. For instance, ψ depends on the relative orientation (θ) of the grain (assumed to be elliptical in shape) with respect to the loading axis, and the width of the grain's minor axis (W), both of which dictate the planes with maximum shear stress. Thus, even for the cases where Λ_d is large, ψ will be restricted to a finite value depending on the projected length of the maximum shear stress plane with respect to θ.

The differences in σ_y of LP1 and LP2 after heat-treatments are also noteworthy. Since those are similar for a given orientation (BD or SD), we discuss only the results obtained in the BD orientation here. After set A heat-treatments in which ST ≤ 1030 °C, significant differences in σ_y of LP1 and LP2 specimens were noted as detailed in the section 3.2. Such differences are due to the varying levels of precipitation of γ'', γ', δ and Laves phases (and their morphologies) in LP1 and LP2. Out of these four microstructural constituents, γ'' is the primary strengthening precipitate followed by γ', δ, and Laves phases [75,76]. The overall strength of an aged IN718 depends on the following mechanisms: solid solution strengthening (σ_{SSS}), strengthening from the precipitates through the coherency strains (σ_c), stress required for shearing the ordered phases (σ_{Sh}) or bypassing them through the Orowan loop mechanism (σ_{Or}). Since the contribution of σ_{SSS} is relatively small, it is reasonable to assume that σ_{fr} (which also includes σ_{SSS}) does not get affected markedly due to the heat-treatments. Consequently, its contribution is taken to be the same in all the heat treated alloys.

Likewise, the strengthening contribution of γ' is considered invariant on the following basis. Due to the sluggish kinetics of γ' precipitation, the size of γ' remains well below ~ 7 nm, which makes it challenging to characterize (both size and V_f) accurately. Moreover, Sui et al [51] and Zhang et al [57] showed the contribution of γ' to the overall strength of the LB-PBF IN718 is ~260 MPa, irrespective of the type of laser scanning parameters or the heat treatment methods. Turning to σ_{Sh}, it was suggested in literature [57,75] that shearing of the precipitate by dislocations is not dominant at the onset of yielding of IN718 and strengthening would

primarily arise from the coherency strains. Hence, the contribution of σ_{Sh} was not considered either as we focus only on σ_y.

An estimate of $\sigma_{c,}$ which arises from the mismatch in the lattice parameters of the precipitates and the matrix, can be made using the strains ε_l around γ″ that are coherent with the matrix as following [75]:

$$\sigma_c = 1.7MG(\sqrt[2]{|\varepsilon_l|^3})\left[\sqrt[2]{\frac{\left(\frac{1}{\Lambda_{\gamma''}}\right)^2 V_f(1-\beta)}{2bR}}\right] \quad (1)$$

where G is the shear modulus (76 GPa), b is the magnitude of the Burgers vector of the γ matrix in Ni based superalloy (0.249 nm [51,75]), $\Lambda_{\gamma''}$ and $2R$ are the aspect ratio, and the major diameter of γ″ precipitates, respectively, and β is ~0.334 as reported in [75]. Several researchers have estimated ε_l to range between 2.3% to 2.8% for the γ″ precipitates after heat treatments similar to HT1 and HT4 [57,75–78]. We assume ε_l to be 2.3% across LP1 and LP2 specimens for estimating σ_c because the size of the coherent γ″ precipitates is similar in both LP1 and LP2 specimens after all the heat-treatments.

From Eq. 1, we note that the two parameters that could significantly affect σ_c are M and $\Lambda_{\gamma''}$. Estimated values of M, made using the EBSD data, are listed in Table 4 for all the heat treated conditions, while representative histograms of M for LP1 (in blue) and LP2 (in red) after heat treatment sets A and B are shown in Figs. 9c and 9d, respectively. Evidently, the weighted average value of M in LP1 continues to be smaller than that of LP2 after set A heat treatments where solutionizing does not eliminate the as fabricated microstructure completely. While M is less than ~2.7 in LP1, it is greater than ~3 for LP2. After set B heat treatments, M for LP1 increases, but only to ~2.94 due to the presence of abnormal columnar grains, as reflected by the spikes in Fig. 9d. This bears a consequence in the prediction of strengthening in set B heat treatment, as will be discussed later.

The contribution from the Orowan looping to strengthening, σ_{Or}, depends on the spacing between the precipitates, which is formulated by Kelly [79] for plate shaped γ″ precipitates as following:

$$\sigma_{Or}^{\gamma''} = \left(\frac{0.85MGb}{2\pi\sqrt{(1-\nu)}}\right)\left[\frac{\sqrt{\Lambda_{\gamma''}V_f}+\left\{\left(\left(\frac{2}{\pi}\right)-\left(\frac{\pi}{2\Lambda_{\gamma''}}\right)\right)\Lambda_{\gamma''}V_f\right\}}{2R\sqrt[3]{\frac{2\Lambda_{\gamma''}}{3}}\left(1-\frac{\pi}{2\Lambda_{\gamma''}}\left\{\sqrt{\Lambda_{\gamma''}V_f}+\left\{\left(\left(\frac{2}{\pi}\right)-\left(\frac{\pi}{2\Lambda_{\gamma''}}\right)\right)\Lambda_{\gamma''}V_f\right\}\right\}\right)}\right]\ln\left(\frac{4R\left(\sqrt[3]{\frac{2\Lambda_{\gamma''}}{3}}\right)}{\pi b}\right). \quad (2)$$

The above equation shows σ_{Or} to be proportional to V_f and $\Lambda_{\gamma''}$ of γ″ precipitates. Additionally, the contribution of δ precipitates to σ_{Or} also needs to be considered. In their case, Kelly proposed that with higher aspect ratio (Λ_δ) and V_f of the rod like precipitates, the extent of strengthening by the looping mechanism can be estimated using the following relation:

$$\sigma_{Or}^{\delta} = \left(\frac{0.85MGb}{2\pi\sqrt{(1-\nu)}}\right)\left[\frac{1}{2R\left(\sqrt[3]{\frac{2}{3\Lambda_\delta}}\right)\left(\sqrt{3}\left\{\sqrt{\left(\frac{\pi}{6\nu_f}\right)}-\sqrt{\frac{2}{3}}\right\}\right)}\right]\ln\left(\frac{2\sqrt{2}R\left(\sqrt[3]{\frac{2}{3\Lambda_\delta}}\right)}{b}\right). \quad (3)$$

According to it, an increasing length ($2R$) of the δ phase will reduce its contribution to the dispersion strengthening.

The Orowan looping is also important in case of Laves phases. As discussed in section 3.2, the spherical Laves phases are present in both the LP1 and LP2 specimens after HT3 heat treatment. Their strengthening contribution depends on the separation between the particles, which can be approximated as $\sqrt{2}\lambda$ since the Laves phase lie on the cell boundaries:

$$\sigma_{Or}^{Laves} = \frac{3MGb}{2\lambda_{Laves}} \quad (4)$$

The overall strengthening contributions from the precipitates along with the Hall Petch contribution due to grain size, d, can be computed as proposed by Yuan *et al.* [80]:

$$\sigma_{Total} = \sigma_0 + \left(\left(\frac{K_{hp}}{\sqrt{d}}\right)^k + \sigma_C^k + \sigma_{Or-\gamma''}^k + \sigma_{Or-\delta}^k + \sigma_{Or-Laves}^k\right)^{1/k} \quad (5)$$

where k = 1.19 for peak aged condition, = 1.15 for underaged or overaged conditions [81], and K_{hp} = 710 MPa.$\sqrt{\mu m}$ for grain boundary strengthening in IN718 [82].

The effect of γ″ on the σ_y (a combination of σ_c, σ_{Sh}, and σ_{Or}) is determined for the respective V_f and $\frac{1}{\Lambda_{\gamma''}}$ of γ″ after all the heat-treatments. Slama et.al [76] and Devaux et.al. [78]

had shown that below a critical major axis size 2*R* and above critical value of $\frac{1}{\Lambda_{\gamma''}}$, coherent and order strengthening is dominant in γ″. This critical value for $\frac{1}{\Lambda_{\gamma''}}$ was found to be ~0.25 for γ″ in present case. Fig. 9b shows the plot of $\frac{1}{\Lambda_{\gamma''}}$ vs. 2*R* of the γ″ precipitate for LP1 and LP2 specimens, with distinct shift in slope when $\frac{1}{\Lambda_{\gamma''}}$ reduces to ~0.25 indicating the end of coherency. This suggests that γ″ formed during HT1 (2*R* ~ 19 nm, $\frac{1}{\Lambda_{\gamma''}}$ ~ 0.34 - 0.4) and HT4 (2*R* ~26 nm, $\frac{1}{\Lambda_{\gamma''}}$ ~ 0.32 - 0.36) are coherent and hardening in these samples will be primarily controlled by σ_c. Due to the coarsening of γ″ in HT2 and HT5 samples, $\frac{1}{\Lambda_{\gamma''}}$ reduces to below 0.25. Thus, σ_{Or} is most dominating contributor to strengthening. While the total proportion of γ″ after HT3 heat treatment is ~10%, fig. S3c shows a bimodal distribution of γ″ with 2*R* varying between 25 nm and 90 nm. This indicates that a combination of different hardening modes may play a role in strengthening after HT3, i.e., DA specimens. The critical size of γ″ above which a transition in the mechanism that dominates strengthening changes from σ_c to σ_{Or} was estimated (from Eqns. 1 and 2) to be 50 nm, for the set of experimentally measured values of $\frac{1}{\Lambda_{\gamma''}}$, V_f and *M*. Even though this finding reveals higher critical size than reported by Zhang et al. (30 – 34 nm), 2*R* = 50 nm correlates to the work on conventionally manufactured Inconel 718 reported by Devaux et al. The measured size distribution of γ″ (Fig. S3) revealed that ~65 and ~75% were coherent (i.e., 2*R* < ~50 nm) in LP1 and LP2, respectively. This means, the remaining ~35 and 25% of σ_y in LP1 and LP2 provide strengthening through the Orowan looping mechanism.

Variations in the estimated σ_y values of HT LP1 and LP2 specimens are displayed in Fig. 9e, where σ_{SSS}, and σ_c from γ′ phase are not included. The experimental and estimated differences between LP1 and LP2's σ_y (all in BD orientation) are plotted in Fig. 9f. A reasonable correlation between the two is seen. On this basis (and that of Fig. 9e), the effect of individual microstructural constituents on σ_y for each alloy and for different HT schedule are listed in Table 6.

4.3. Ductility of the heat treated alloys

The orientation relation between the orthorhombic ($D0_a$) δ precipitates and the γ matrix is $\{111\} < \bar{1}01)>_\gamma$// (010) $[100]_\delta$ [83]. Depending on the four closed pack habit planes of γ, 12 different variants of δ are possible, which fall into 3 different subsets [40,83]: $[1\bar{1}0]_\gamma$ // $[100]_\delta$,

$[111]_\gamma$ // $[010]_\delta$, and $[11\bar{2}]_\gamma$ // $[001]_\delta$. As discussed before, LP1 and LP2 in the as-built state exhibit <100> and <110> textures along BD, respectively. These textures remain even after HT, irrespective of the heat treatment schedule employed. Such dominant textures can result in a preferential growth of δ, depending on the variant selection with respect to γ-matrix. For example, in the forged discs of IN718, cells of δ can form in a repeated arrays with respect to the texture of the formed parts, i.e., direction of the forming [84,85]. To illustrate a similar arrangement of δ in the present case, the microstructures of LP1 after HT1 are shown in Figs. 10a to f. A closer examination of grains with <100> build texture in Figs. 10c and e reveals that δ are oriented at ± ~45° to BD and consequently to the loading axis. In contrast, Figs. 10d and f show that δ is aligned at either ~0 or 90° to the loading axis in a grain with the <110> orientation. The idealized alignment of δ needles with respect to tensile stress after set A heat-treatments, which only led to the formation of δ, are illustrated schematically in Figs. 11a and b. Fractography of HT1 samples revealed that the δ precipitates gets preferentially ruptured (see Figs. 11c to f) by shearing of the γ – δ interface, leaving traces of δ on the fracture surfaces of both the LP1 and LP2 specimens. The Nb rich regions shown in the fracture surface (see Fig. 11f) are traces of δ-phase.

The critical strain (ε_c) required to nucleate a void on the γ – δ interface can be estimated as [86,87]:

$$\varepsilon_c = \sqrt{\frac{(G_\gamma^{eff}{}_\gamma b_\gamma + G_\delta^{eff} b_\delta)}{1.67 E_\delta^{eff} w_\delta}} \tag{6}$$

where G_γ^{eff} and G_δ^{eff} are the shear modulus; b_γ and b_δ are the Burgers vectors of the dislocation inside γ-matrix and δ, respectively. E_δ^{eff} is the effective Young's modulus and w_δ is the effective width of δ along the loading direction. The values of b_γ and b_δ are 0.249 and 0.256 nm, respectively [88,89]; The average length and width of δ in HT1 are ~1 μm and ~50 nm, respectively, in both LP1 and LP2 specimens. The elastic theory of orthotropic material was used to compute the E_δ^{eff}, G_δ^{eff} of δ and G_γ^{eff} in both the cases assuming plane stress conditions. The following transformation relations (equations 7 and 8) expressed in terms of elastic constants, E_1, E_2, G_{12}, ν_{12}, ν_{21} and θ. Here, θ is the angle between the loading axis and crystal structure axis along <100> and, m and n are cosθ and sinθ, respectively [90]:

$$\frac{1}{E_{\delta,\gamma}^{eff}} = \frac{m^2}{E_1}(m^2 - n^2\nu_{12}) + \frac{n^2}{E_2}(n^2 - m^2\nu_{21}) + \frac{m^2 n^2}{G_{12}}, \tag{7}$$

$$\frac{1}{G_{\delta,\gamma}^{eff}} = \frac{4m^2n^2}{E_1}(1+\nu_{12}) + \frac{4m^2n^2}{E_2}(1+\nu_{21}) + \frac{(m^2-n^2)^2}{G_{12}}. \quad (8)$$

The values of elastic constants of δ and γ-matrix were taken from Gaillac *et al.* and are estimated (Table S4) by solving equations S.1, S.2, and S.3 of supplementary information [91]. G_γ is assumed to be similar to pure Ni because the γ-matrix around the δ would be almost free of Nb, Mo in the solid solution [92]. In case of LP1 where δ is aligned at 45° to BD and the loading axis, $\theta = 45°$. Substituting the values of elastic constants in equation 7 and 8 yields E_δ^{eff} and G_δ^{eff} for LP1 specimens to be ~162 and ~100 GPa, respectively. For LP2, in which the precipitates are aligned to the loading axis (θ = 0°), E_δ^{eff} and G_δ^{eff} can be directly found from the stiffness matrix of δ to be ~238 and ~52 GPa, respectively. Similarly, G_γ^{eff} can be computed for γ matrix loaded in <100> direction (θ = 0°), and <110> directions (θ = 45°), to be ~138 and 58 GPa, respectively.

After substituting these values in Eq. 6, ε_c was estimated to be ~6.65% for LP1 and ~4.25% in LP2 specimens. These calculations illustrate that for the same level of applied stress, voids at γ – δ interface of LP2 are likely to nucleate significantly earlier than in LP1. Additionally, the number of potential nucleation sites for such voids is higher in LP2 as A_f of δ is ~10% (compared to ~8.5% in LP1). Once a void nucleates at the interface, it is much easier for them to propagate in LP2 specimens due to their <110> texture, which was also observed in as-built LP2 specimens. Ductility of LP2 specimens gets lowered markedly after set A heat treatments (by ~60% as compared to only ~19% in case of LP1 specimens because of this weakness arising from the γ/δ interface in them with δ in the microstructure. Since δ phase was not observed in alloys subjected to set B heat-treatments, their ductility does not vary. These observations highlight the importance of initial microstructure in LB-PBF IN718, considering the unusual ductility variation in the presence or absence of δ in LP2.

4.4. Implications

The following are the implications of the results obtained in the present work, in terms of designing the heat treatment strategies for LB-PBF IN718 to achieve good tensile properties. This is particularly relevant in the case of alloys such as IN718 since they are rarely used in the as built condition and almost always are heat treated so as to enhance the strength through precipitation hardening. In such a context, it is necessary to realize that the as built microstructural features can get partially or completely modified after heat treatment. Hence,

attractive mechanical properties in as built condition alone does not suffice in developing LB-PBF Inconel 718.

As illustrated in this study, the microstructural features in as fabricated condition have legacy effect on the evolution of grains, precipitates, and tensile properties after the heat treatment. Therefore, the intended application of the part manufactured should be kept in mind while optimizing the processing parameters, i.e., they should be dictated not just by highest as built strength and/or lowest porosity. For example, finer grains are desirable for structural alloys in fatigue intensive applications. Thus, processing parameters which results in high stored strain energy leading to fully recrystallized grains after the heat-treatment would be desirable. Similarly, while designing microstructure requiring preferred orientation and columnar grains over large length scales, the process parameters should be tuned to limit the fraction of low angle grain boundaries in order to avoid heterogenous microstructure after heat treatment. Additionally, the processing parameters should be designed with an eye on the precipitate evolution due to scheduled post-build heat treatments as the current study shows how they affect strength and ductility. In this manner, post heat treatment microstructural evolution and the mechanical properties should be considered to optimize the process parameters for LB-PBF Inconel 718. While this study shows the cascading effect of as built microstructures on room temperature tensile properties, further investigation is needed to understand its effect at high temperatures.

5. Summary and Outlook

A comprehensive experimental investigation into the microstructures and the tensile properties of Inconel718 alloy processed using two different sets of LB-PBF parameters were performed, with particular emphasis on the influence of as-built microstructures on the properties of heat treated ones. Five heat treatments were conducted by selecting solutionizing temperature above and below the solvus of δ (1030 °C) to vary the type, proportion, size, and coherency of the strengthening precipitates. The experimental results and analyses show that in the as built condition, the differences in effective mean free paths for the dislocation motion within the solidification cellular structures can be utilized to rationalize the variation in strengths.

Single or double step aging treatments with the solutionizing temperature below the δ solvus preserve the as-built grain morphology. Although, anisotropy cannot be completely eliminated by such heat treatments, it is significantly smaller in the alloy with a lower proportion of LAGBs. Since precipitates such as γ″ and δ preferentially nucleate on certain

crystallographic planes, retention of the as built texture has a cascading effect on tensile strength and ductility. Highly acicular δ phase act as sites of void nucleation due to strain mismatch with the matrix. The anisotropic nature of the δ phase makes ductility depend on built orientation with respect to the loading direction. Heat treatment at 1100 °C completely dissolves the cellular structure, leading to a good combination of strength and ductility.

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Table 1. Processing parameters used for the two alloys examined in the present study.

Parameter	LP1	LP2
Power (W)	180	140
Scan speed (mm/s)	800	1200
Scan strategy	Island	Island
Scan rotation between layers	0°	90°
Hatch spacing (μm)	105	90
Layer thickness (μm)	25	20
Volumetric energy density (J/mm^3)	~89	~65
Laser spot size (μm)	130	30
Melt pool width (μm)	110-120	35-40
Melt pool depth (μm)	40-50	35-50
Overlap %	16-17	0
Powder condition	reused	new

Table 2. Details of the heat treatment schedules employed.

	Solutionizing temperature (°C)	Cooling method	First aging conditions: temperature (°C) – time (h)	Cooling rate (°C/h)	Second aging conditions: temperature (°C) -time (h)
HT1	960	Water Quench	718 – 8h	50	621 - 8h
HT2	1030	Water Quench	790 – 8h	-	-
HT3	-	-	760 -10h	50	649 - 8h
HT4	1100	Water Quench	718 - 8h	50	621 - 8h
HT5	1100	Water Quench	760 - 10h	50	649 - 8h

Table 3. Chemical compositions (in wt.%) of the fabricated alloys and the powder.

Material	Ni	Fe	Cr	Nb	Mo	Ti	Al
LP1	52 ± 0.4	17.99 ± 0.11	19.24 ± 0.14	5.36 ± 0.18	3.26 ± 0.16	0.97 ± 0.06	0.6 ± 0.04
LP2	51.33 ± 0.25	17.8 ± 1.48	19.54 ± 1.1	5.40 ± 0.68	3.34 ± 0.44	0.93 ± 0.08	0.55 ± 0.06
Powder	50 – 55	18 – 20	17 – 21	4.75 – 5.5	2.8 – 3.3	0.65 – 1.15	0.2 – 0.8

Table 4. Grain sizes and aspect ratios in different alloys.

Heat treatment	Alloy	Grain size (μm)	Grain aspect ratio (Λ_d)	Weighted average Taylor factor (*M*)
AB	LP1	18 ± 13	3.49	2.62
	LP2	12 ± 10	2.58	3.17
HT1	LP1	25 ± 20	3.37	2.71
	LP2	15.2 ± 20	2.69	3.03
HT2	LP1	74 ± 53	6.98	2.60
	LP2	37 ± 30	3.40	3.10
HT3	LP1	56 ± 38	5.34	2.56
	LP2	36 ± 29	2.50	3.09
HT4	LP1	44 ± 30	4.57	2.94
	LP2	33.1 ± 41	2.80	3.00
HT5	LP1	55 ± 41	4.36	2.70
	LP2	33.2 ± 29	2.47	3.08

Table 5. Tensile properties of various alloys. Values in parentheses are percentage increases in the respective properties in the as-built condition.

Condition	Alloy	Build orientation			Scan orientation		
		σ_y (MPa)	σ_u (MPa)	e_f (%)	σ_y (MPa)	σ_u (MPa)	e_f (%)
AB	LP1	618 ± 22	952 ± 9	29.5 ± 3	723 ± 4	1010 ± 1.5	27.9 ± 2.5
	LP2	780 ± 32	1069 ± 7.5	18.6 ± 0.5	892 ± 7.6	1153 ± 9.2	23 ± 0.3
HT1	LP1	1143 ± 8 (85)	1391 ± 6 (46)	24 ± 0.8 (-19)	1219 ± 26 (68)	1421 ± 10 (40)	15 ± 0.4 (-47)
	LP2	1248 ± 2 (60)	1400 ± 18 (31)	7.4 ± 0.4 (-60)	1291 ± 8 (45)	1485 ± 25 (29)	11 ± 6.7 (-50)
HT2	LP1	900 ± 23 (46)	1207 ± 5 (27)	30 ± 1 (0.2)	1007 ± 23 (39)	1264 ± 2.5 (25)	22 ± 1.8 (-21)
	LP2	1049 ± 20 (34)	1220 ± 20 (14)	6.3 ± 2.2 (-66)	1111 ± 3 (25)	1344 ± 15 (17)	16 ± 1 (-30)
HT3	LP1	1103 ± 38(78)	1382 ± 12 (45)	23 ± 2 (-22)	1213 ± 22 (68)	1440 ± 10 (42)	14 ± 0.5 (-50)
	LP2	1283 ± 19 (64)	1448 ± 29 (35)	7 ± 2.4 (-61)	1329 ± 9 (49)	1541 ± 4 (34)	12 ± 0.5 (-49)
HT4	LP1	1156 ± 7 (87)	1359 ± 15 (46)	22 ± 2 (-19)	1246 ± 36 (72)	1371 ± 9.6 (36)	21 ± 7 (-23)
	LP2	1137 ± 41 (46)	1387 ± 9 (30)	19 ± 4 (2.5)	1188 ± 7 (33)	1441 ± 11 (25)	27 ± 1 (15)
HT5	LP1	1137 ± 11 (84)	1352 ± 2 (42)	24 ± 2.6 (-18.6)	1223 ± 12 (69)	1418 ± 1.2 (40)	22 ± 1 (-21)
	LP2	1228 ± 29 (57)	1454 ± 12 (36)	19 ± 1 (2.4)	1216 ± 11 (36)	1468 ± 18 (27)	21 ± 6.6 (-10)

Table 6. Effect of the microstructural constituents on the strengthening behaviour of LB-PBF Inconel 718 along BD orientation.

Condition	$\Delta\sigma_y^{LP2-LP1}$ (MPa)		Remarks
	Experimental	Predicted	
HT1	105	130	• V_f of γ'' is similar for both conditions, therefore, a difference of 99 MPa in σ_c arise from variation in $\frac{1}{\Lambda_{\gamma''}}$ and M, i.e., $\frac{1}{\Lambda_{\gamma''}}_{LP2}$ (~0.4) > $\frac{1}{\Lambda_{\gamma''}}_{LP1}$ (~0.33) and M_{LP2} (~ 3.03) > M_{LP1} (~2.71). • σ_{or} caused by δ precipitates in both the specimens is similar since $2R_\delta > 1$ μm for both the conditions. • Relatively high σ_{HP} compared to other HT specimens was observed in both LP1 (102 MPa) and LP2 (135 MPa) because the grain growth was restricted by the formation of grain boundary δ.
HT2	149	133	• Overaging of γ'' resulted in loss of coherency, thus, σ_c ~ 0. • γ'' provides effective strengthening through Orowan looping, which is 69 MPa higher in case of LP2 because M_{LP1} (~ 2.60) < M_{LP2} (~ 3.10). • Severe grain growth led to negligible grain boundary strengthening σ_{HP} ~25 MPa in LP1 in contrast to ~73 MPa in LP2.
HT3	180	181	• Bimodal distribution of γ'' in the range of 25 – 90 nm indicates presence of both σ_c and σ_{Or}. • Difference in σ_c between LP1 and LP2 is relatively small (29 MPa) as the size of coherent precipitates is in the same range of 30 ± 4 nm. • σ_{Or} in the LP2 specimen is 49 MPa higher compared to LP1, because the size of γ'' is 77 ± 14 nm in case of LP1 compared to 56 ± 9 nm for LP2 specimens. • Although the difference in V_f of δ in LP1 (~5.4%) and LP2 (6.5%) is not significant, the difference in σ_{or} is amplified to 47 MPa due to variation in M of LP1 (~ 2.56) and LP2 (~3.09) • The difference in σ_{Or} from Laves phases is ~75 MPa because of higher V_f in LP2 specimens. • Severe grain growth led to negligible σ_{HP} ~27 MPa in LP1 compared to ~77 MPa in LP2.

HT4	5	23	• V_f and 2R (26 nm) of γ'' is equal for both LP1 and LP2 case, which should give rise to a similar σ_c. However, the predicted difference of 23 MPa in σ_c arises from variation in M, i.e., M_{LP2} (~ 3.0) > M_{LP1} (~2.94). • If the abnormal columnar grains in LP1 specimens are ignored, the value of M_{LP1} will tend to 3, thus, resulting in a small difference in σ_c ~10 MPa. • Overall grain size of 44 ± 30 μm in LP1 and 33.1 ± 41 μm in LP1 led to a small difference of 21 MPa in σ_{HP}.
HT5	91.5	87	• Both V_f and 2R (62 nm) of γ'' are similar in LP1 and LP2, resulting in similar strengthening by σ_{Or}. • Predicted difference of 87 MPa in σ_c arises from variation in M, i.e., M_{LP2} (~ 3.08) > M_{LP1} (~2.70). • In the likelihood that the extracted tensile samples of LP1 have large proportion of abnormal columnar grains, the predicted difference in σ_y matches with test. • Overall grain size of 55 ± 41 μm in LP1 and 33.2 ± 29 μm in LP1 led to a small difference of 25 MPa in σ_{HP}.