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Surface treatment impact on fatigue life at 550°C of the as-built Inconel 718 manufactured by laser-powder bed fusion

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7 Abstract

4

This article compares the impact of surface modification on fatigue life at 550 °C of the Inconel 718 manufactured by laser-powder bed fusion. The surface conditions are as-built, as-machined, shot-peened, oxidized, and combinaisons of those. Surface roughness, microhardness, and residual stress measurements, as well as fractographic analyses are performed to interpret the fatigue results. Fatigue tests show that as-built samples have higher fatigue lifetime than as-machined samples. Moreover, shot peening increases fatigue lifetime. Finally, an oxidation treatment at 550 °C during 41.6 days does not affect fatigue life.

⁸ Keywords: additive manufacturing, laser-powder bed fusion, Inconel 718,

⁹ fatigue, shot peening, oxidizing, elevated temperature.

10 1. Introduction

¹¹ Metal additive manufacturing is a disruptive technology that is likely to ¹² replace some traditional manufacturing processes. Among the many available

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additive manufacturing processes, the present study focuses on the laser-power
bed fusion (L-PBF) process.

Metal additive manufacturing offers numerous advantages including faster 15 and lower-priced production [1] for more complex geometries [2]. The working 16 principle of these processes consists in adding feedstock material layer upon 17 layer until shaping the desire piece. However, this process creates undesirable 18 high surface roughness [3], porosity [4], and tensile residual stresses [5]. 19 These side effects involve that as-built (AB) test pieces have drastic inferior 20 fatigue life in comparison with that of the conventional counterparts [3, 21 6]. In materials science, fatigue life characterizes the ability of a material 22 submitted to repeatedly loading to withstand without fracturing. Fatigue 23 life is a key parameter in mechanical design, and is currently what disqualify 24 additive manufacturing pieces from being used for critical application. As 25 a consequence, fatigue life of additive manufactured pieces is a hot research 26 topic, with the aim of providing reliable printed structures. 27

28

This study focuses on the L-PBF processing of Inconel 718 (IN718) alloy. This nickel-iron based superalloy is widely used, in the form of cast and wrought products, in the aerospace, nuclear and submarine industries for high temperature applications [7]. In recent years, with the increasing maturity of additive manufacturing processes, this alloy has been examined closely in the context of Additive Manufacturing. The microstructure of IN718 is composed of a face-centered cubic matrix, which is strengthened with

precipitates γ " and γ ' and fine micrometric precipitates of δ phase located at 36 grain boundaries [4, 8]. Additive manufactured IN718 contains unfavorable 37 compounds regarding mechanical properties such as laves phase and a large 38 amount of acicular delta precipitates decorating the grain boundaries [4, 8]. 39 These detrimental morphology and location of the δ phase are caused by the 40 rapid cooling rate inherent in additive manufacturing process. Moreover, the 41 saturation of δ phases reduces the amount of Nobium available to form the 42 γ " strengthening phase, leading to a potential decrease in the strength of the 43 material. [4, 8]. As a consequence, IN718 pieces additively manufactured are 44 usually heat treated after manufacturing to release potential residual stresses, 45 to reduce the amount of δ phase, and to modify the morphology of these δ 46 precipitates. These post additive manufacturing heat treatments aim also at 47 maximizing the γ' and γ'' strengthening precipitates [4, 8]. 48

49

At room temperature, several authors [3, 9–13, 13, 14] have compared 50 fatigue properties of IN718 pieces additively manufactured with their wrought 51 counterpart. Some reports [10-13] showed that low cycle fatigue (LCF) 52 properties of IN718 pieces additively manufactured in an as-machined (AM) 53 or as-polished (AP) condition were comparable to those of the wrought 54 counterpart. However, the conclusions regarding the comparaison of the 55 fatigue properties in the high cycle fatigue (HCF) regime differ according 56 to the studies. On the one hand, some results showed fatigue properties of 57 additively manufactured IN718 parts in AM or AP conditions comparable 58

to those of wrought parts [3, 9, 12–14], but on the other hand, some studies showed inferior properties for additively manufactured parts. [9, 12]. The divergent conclusions were likely due to the different heat treatments and surface finishes employed by the authors.

In particular, Lambert [9] compared the fatigue results of IN718 parts produced 63 by additive manufacturing, all subjected to an identical heat treatment but 64 featuring different surface treatments, namely, low stress grinding, bead 65 blasting, and micromachining processes. The results showed that the samples 66 having a low stress grinding surface provided HCF fatigue results comparable 67 to those of a wrought IN718 sheet, while the other surface treatments provided 68 lower fatigue results. Other studies have shown the importance of the surface 69 condition of additively manufactured parts in IN718 on fatigue life [3, 13, 15]. 70 Indeed, these authors have shown experimentally that the fatigue life of 71 the AB condition was significantly lower than that of the AM and AP 72 conditions. For example, Kelley [15] observed that the fatigue lives of AM 73 IN718 samples were 10 and 1000 times higher than those of the AB state 74 in the LCF and HCF regimes, respectively. Such fatigue lifetime difference 75 could partially be attributed to the AB pieces surface roughness, which was 76 about 4 [15] and 10 [3] times higher in average than the AM piece surface 77 roughness. Similar observations regarding the enhancement of fatigue life 78 after machining and polishing were obtained at 427 °C [16] and at 650 °C 79 [16, 17]. However, conclusions regarding those results should be carefully 80 analysed, as the metallurgical state and the materials quality were highly 81

⁸² dependent on the scanning strategies.

High temperature comparison of the fatigue properties of additively manufactured 83 IN718 parts with their wrought counterparts remains rare. Sui *el al.* [18] and 84 Song et al. [19] compared the HCF properties of additively manufactured 85 IN718 parts in AP condition with those of their wrought counterparts at 86 650 °C. Sui *el al.* [18] observed a higher fatigue lifetime for additively 87 manufactured IN718 parts than for wrought parts at low stress amplitudes, 88 but a shorter fatigue life for additively manufactured IN718 parts at high 89 stress amplitudes. However, Song et al. [19] observed an opposite trend, 90 with similar fatigue lifetime in HCF for the additively manufacturing and 91 the wrought materials. Sui *el al.* [18] performed a direct aged precipitation 92 treatment on the fabricated samples, while Song *et al.* [19] performed a 93 solid solution annealing prior to double aging, likely resulting in a higher 94 proportion of γ' and γ'' precipitates. The two distinct heat treatments 95 employed by the authors could explain the differing conclusions regarding 96 the HCF performance of these additively manufactured samples. 97

In conclusion, in the HCF regime, fatigue properties of IN718 pieces additively manufactured can be similar to those of the wrought IN718 counterpart under particular conditions. In the LCF regime, fatigue properties at room and high temperatures were similar for additive manufactured and wrought materials. Surface finish and heat treatment appeared to strongly influence the fatigue performance of the IN718 additively manufactured. More specifically, several studies [3, 9, 13, 15–17] suggested that AB pieces should be machined or ¹⁰⁵ polished to improve fatigue performance. Finally, comparaisons of Song *et*¹⁰⁶ *al.* [19] and Sui *el al.* [18] studies suggested that annealing before aging the
¹⁰⁷ AB IN718 improved fatigue performance. Herein, the studied samples were
¹⁰⁸ therefore annealing before aging.

109

Shot peening is a surface treatment commonly used to improve fatigue life 110 [20, 21]. The main purpose of shot peening consists in impacting a surface 111 with high velocity shots to introduce compressive residual stress, hardening, 112 and potential microstructural changes [20-22]. In addition, this process 113 annihilates the pores near the surface that can be introduced during casting 114 or additive manufacturing [23-25]. However, the shot peening treatment 115 modifies the surface roughness, which may result in a decrease of fatigue 116 properties [20, 21]. 117

The effect of shot peening on fatigue performance of test pieces additively 118 manufactured was studied by Wycisk *et al.* [26], Bagherifard *et al.* [27], and 119 Wood et al. [28] for Ti-6Al-4V, AlSi10Mg, and 316L materials, respectively. 120 These studies showed that shot peening treatements improved fatigue life of 121 AB test pieces. In particular, Bagherifard *et al.* [27] and Wood *et al.* [28] 122 directly shot peened AB test pieces. These shot peening treatments resulted 123 in a large reduction in surface roughness, with an arithmetic mean value of 124 roughness that was 2 [27] and 4 [28] times less than that of the AB surface. 125 This reduced roughness is likely to be partly responsible for the improved 126 fatigue properties. 127

In regard to IN718 in additive manufacturing, the impact of shot peening on fatigue life was studied at room temperature by Ardi *et al.* [29]. The authors observed that shot peening with S230 media at an Almen intensity of 18A and 200% coverage increased the fatigue life of HCF when compared to AP samples.

133

Shot peening is traditionally used as a surface treatment to increase fatigue life, although it increases surface roughness which deteriotes fatigue life. In addition, poor fatigue property of additively manufactured structures is the major hindrance for structural applications. Recent studied [26–29] have shown that shot peening decreased surface roughness of additively manufactured samples. Therefore, shot peening appears as a perfect candidate to increase fatigue property of AB additively manufactured material.

The goal of this paper was to verify that shot peening IN718 AB samples was sufficient to obtain at elevated temperature HCF lifetime values as good as those with an AM surface finish. Since IN718 material is usually used for high temperature application effect of oxidation on fatigue life was also studied besides shot peening. For this purpose, AB and AM samples were shot peened and/or oxidized prior to be fatigue testing.

7



Figure 1: Scanning strategy used to print the fatigue specimens.

¹⁴⁷ 2. Material and experimental details

148 2.1. Material

The studied material was an Inconel 718 nickel superalloy additively 149 manufactured by a L-PBF process. Raw material powder was provided by 150 Praxair with a diameter ranging from 15 to 45 µm. Samples were manufactured 151 using an EOS M280-5 3D printer in a nitrogen inert gas atmosphere. The 152 machine setup is confidential. Specimens were printed following a contour 153 hatch scanning strategy: the outward contours of the studied specimens 154 were first laser printed, then the enclosed surfaces were filled in using a 155 hatch strategy, as shown in Figure 1. The layer thickness was 40 µm and the 156 scanning direction rotated from layer to layer with an angle of 67°, as shown 157 in Figure 1. 158

159 2.2. Heat treatments

The residual stresses introduced during the L-PBF process were thermically relaxed to prevent specimens from cracking. Therefore, prior removing from the support structure, the printed specimens were heat treated at $975 \,^{\circ}C$ during 30 minutes.

164

The AB microstructure after thermal relaxation contained a high density of δ phases, that limited the potential amount of strengthening γ'' particles precipitation during aging [4]. As a consequence, the printed specimens were heat treated at 1080 °C during 1 hour to dissolve the δ phases, and double aged at 710 °C and at 620 °C both during 8 hours in order to induce the precipitation of the γ' and γ'' phases.

171

172 2.3. Microstructure

Figure 2 shows the heat treated microstructure in the plane perpendicular 173 to the building direction (BD), which was the loading direction. Figure 2a 174 presents an inverse pole figure (IPF) map combined with an image quality 175 (IQ) map from electron backscatter diffraction (EBSD) data. This image 176 shows a duplex grain size distribution with finer grains at the surface and 177 within the sample along the scanning direction (SD). These finer grains 178 were the consequence of the high cooling rate induced by the contour hatch 179 scanning strategy. Figure 2b is a scanning electron microscope (SEM) image 180 at low magnification. This figure reveals a high density of porosities within 181 the material, with a large amount of coarse lack of fusions known to have a 182 detriment effect on fatigue life. Finally, a residual amount of the δ phases 183

located at the grain boundaries was observed at high magnification, as shown
in Figure 2c. At first slight, these precipitates were unlikely to have a
detrimental effect on fatigue.

187 2.4. Tensile test

A tensile test was performed in order to provide an idea of the mechanical 188 tensile properties of the printed material. The tensile specimen was loaded in 189 the BD up to a total deformation (ϵ_{tot}) of 2% using a MTS 810 servo-hydraulic 190 test machine. A MTS 652.51C.04 extension ter having a gauge length of 191 $12 \,\mathrm{mm}$ was used to experimentally measure ϵ_{tot} . The sample was heated at 192 $550\ensuremath{\,^\circ C}$ using a MTS 653 furnace. The geometry of the tensile specimen was 193 identical to the as-machined fatigue specimen geometry, which is shown in 194 Figure 4c. 195

196

The strain rate has been modified during testing to quantitatively evaluate the material's viscosity. For ϵ_{tot} ranging from 0% to 1% and from 1.5% to 2.0%, a strain rate of 10^{-3} s⁻¹ was imposed, while for ϵ_{tot} ranging from 1% to 1.5% the strain rate was set to 10^{-5} s⁻¹.

201

Table 1 summarizes the tensile properties extracted from the tensile curve shown in Figure 3, and provides the equivalent tensile properties for a conventional IN718 [30]. This table shows that the studied IN718 was similar at high temperature to a conventional counterpart in terms of tensile

Table 1: Tensile properties of the studied material at 550 °C compared to the litterature. E, σ_y , and σ_u are the Young's modulus, the yield strength, and the tensile strength, respectively.

| | Temperature | E | $\sigma_{\rm y}$ at 0.2% offset | $\sigma_{\rm u}$ |
|---------------------------|---------------|-------|---------------------------------|------------------|
| | $[^{\circ}C]$ | [GPa] | [MPa] | [MPa] |
| Studied material | 550 | 173 | 1130 | 1224 |
| Conventional IN718 $[30]$ | 538 | 172 | 1068 | 1275 |

²⁰⁶ properties.

207

Figure 3 presents the tensile test results. This figure shows that viscosity had a minor impact on the material behavior because the stress response was similar at $\epsilon_{tot} = 1\%$ for both strain rates. In addition, a serrated yielding and an abrupt stress response were observed on the tensile curve. This was most likely due to a dynamic strain aging phenomenon, also called Portevin-Le Chatelier effect. This effect has been broadly reported at hight temperature for conventional IN718 [31, 32].

215 2.5. Surface conditions

Several surface conditions were studied. The baseline conditions were as-built (AB) and as-machined (AM). A number of the AB and AM samples were shot peened (SP) leading to two extra conditions: AB-SP and AM-SP. Similarly, a number of the AB and AM samples were oxidized. The corresponding surface conditions were named AB-Ox and AM-Ox. Finally, a number of the shot peened samples were oxidized. The corresponding surface conditions

| | As-built | As-machined |
|--------------------------|----------|-------------|
| Baseline | AB | AM |
| Shot peened | AB-SP | AM-SP |
| Oxidized | AB-Ox | AM-Ox |
| Shot peened and oxidized | AB-SP-Ox | AM-SP-Ox |

Table 2: Studied surface conditions and the corresponding acronyms.

were named AB-SP-Ox and AM-SP-Ox. Table 2 summarizes the eight studied surface conditions along with their acronyms used in the present study. The shot peening and oxidizing conditions are described in Section 225 2.6.

226

227 2.6. Shot peening and oxidizing conditions

Conventional shot peening was performed by an external company, namely, IRT M2P. Samples were shot peened with cut wire 400-700 media, with a nominal Almen intensity of F12A millimeters, and a coverage of 150%. Media were controlled following VDFI 8001 standard [33]. Almen intensities were measured as per SAE J443 standard [34]. Finally coverage were estimated under an optical microscope.

234

Oxidized samples were heated under constant temperature at 550 °C during 41.6 days (1000 hours) in an air environment. These oxidizing conditions provided a nanometric oxide layer, and therefore did not affect surface roughness.

238 2.7. Surface roughness measurements

Surface roughness may affect fatigue life results. As a consequence, surface roughness of AB, AB-SP, AM, and AM-SP samples were characterised in the building direction (BD) using a Tribotechnic profilometer. Four profiles were measured per condition at a recording speed of $500 \,\mu m \cdot s^{-1}$. Arithmetic average roughness (R_a) were extracted from the measured profiles.

244 2.8. Microhardness measurements

Hardening and residual stress may impact fatigue life results. Microhardness
profile provides an indication of hardening and residual stress levels within
the material. Thus, in-depth microhardness profiles were measured for the
studied surface conditions using a Buehler micromet microhardness tester at
a force of 100 g.

250 2.9. Residual stress measurements

As a complement to the microhardness measurements, surface residual stresses were measured on AM, AB, AB-SP, and SP-Ox samples.

The residual stresses were determined by the x-ray diffraction technique, in which the strain in the crystal lattice was measured, and the residual stress producing the strain was calculated, assuming a linear elastic distortion of the crystal lattice. The measurements were performed in a Panalytical Empyrean X-ray diffractometer equipped with a $\theta - \theta$ goniometer and Cu-K α ($\lambda =$ 1.5406 Å) as incident radiation, operated at 45 kV and 40 mA. The incident beam was focused, using 5 mm and 1/4° apertures, on the (420) reflection of the face centered cubic (fcc) Ni-Cr phase ($2\theta = 147.19^{\circ}$) and the in-plane stress was measured by applying instrumental ω -tilts and performing $\theta - \theta$ scans, in three different directions $\varphi=0^{\circ}$, 45°, 90°.

264

The analysis was based on the $\sin^2 \psi$ method, for which the lattice spacings 265 d were measured at a total of 13 ψ -angles between -50° and 50° (positive and 266 negative ψ -tilts) that correspond to $\sin^2\psi$ values from 0 to 0.6 with steps 267 of 0.1. The stress tensor was calculated by performing a multidirectional 268 stress analysis using the Panalytical's Stress version 2.3 software. Biaxial 269 plane-stress conditions were assumed, involving zero normal stress to the 270 specimen surface (σ_{33}^S) . The peak position was found by fitting a Pearson VII 271 profile shape function to the data, and the following intensity corrections were 272 applied on the measured scans: background subtraction, Lorentz-polarization, 273 and K2-stripping. Absorption correction was also applied even though the 274 impact on the intensity was insignificant. 275

276

The stresses were determined by a least square fit of the elliptical $\sin^2 \psi$ which related the deformation $\epsilon_{\varphi\psi}^{hkl}$, measured by X-ray diffraction, and the components of the stress tensor in the specimen reference frame as follows:

$$\epsilon_{\varphi\psi}^{hkl} = \frac{d_{\varphi\psi}^{hkl} - d_0^{hkl}}{d_0^{hkl}} = \frac{1}{2} S_2^{hkl} (\sigma_{\varphi}^S - \sigma_{33}^S) \cdot \sin^2 \psi + \frac{1}{2} S_2^{hkl} \tau_{\varphi}^S \sin \psi + \epsilon_{\varphi0^\circ}^{hkl}$$
(1)

where $\sigma_{\varphi}^{S} = \sigma_{11}^{S} \cdot \cos^{2}\varphi + \sigma_{12}^{S} \cdot \sin(2\varphi) + \sigma_{22}^{S} \cdot \sin^{2}\varphi$ is the normal stress, $\tau_{\varphi}^{S} = \sigma_{13}^{S} \cdot \cos\varphi + \sigma_{23}^{S} \cdot \sin\varphi$ the shear stress, $\epsilon_{\varphi 0^{\circ}}^{hkl} = \frac{1}{2}S_{2}^{hkl}\sigma_{33}^{S} + S_{1}^{hkl}(\sigma_{11}^{S} + \sigma_{22}^{S} + \sigma_{33}^{S})$ the strain, σ_{ii}^{S} the normal stress tensor components, and σ_{ij}^{S} the shear stress tensor components.

The slope provided an estimation of residual stress assuming the material X-ray elastic constants $S_1^{420} = -1.48 \text{ TPa}^{-1}$ and $\frac{1}{2}S_2^{420} = 6.61 \text{ TPa}^{-1}$ for the Ni (420), as retrieved from the database of the analysis software, and d_0 the unstressed lattice spacing [35, 36].

285 2.10. Fatigue tests

The AB samples were directly printed on the built plate in the BD, while 286 the AM samples were extracted from printed cylinders also in the BD, as 287 schematically shown in 4a. The geometry of the AB, AB-SP, and AB-SP-Ox 288 samples is shown on Figure 4b. The geometry of the AM, AM-SP, and 289 AM-SP-Ox samples is shown on Figure 4c. The fatigue tested volume of 290 both geometries were different. In fact, the AB samples had a tested volume 291 1.8 higher than the AM samples, leading to a potential size effect on the 292 fatigue results. However, the consistency in the crack initiation causes, and 293 the fact that AB samples with higher tested volume had a better fatigue 294 lifetime than AM samples, suggested that the size effect did not impact the 295 present fatigue results. 296

297

Axial fatigue tests were performed in the BD under constant stress amplitude

at a stress ratio (R) of 0.1 using a MTS 810 servo-hydraulic test machine at 299 10 Hz frequency. Samples were heated at 550 °C using a MTS 653 furnace. 300 Three samples were fatigue tested per condition, except for the AM condition 301 where five samples were fatigue tested to approximatively determine the 302 maximum stress required to attain 5×10^4 cycles. This number of cycles 303 were targeted to limit potential fatigue runout after shot peening. Finally, 304 fractured samples were analysed under SEM to determine the crack initiation 305 causes. 306

307



Figure 2: Microstructure of the studied material in the plane perpendicular to the BD. (a) Inverse pole figure (IPF) map combined with an image quality (IQ) map from electron backscatter diffraction (EBSD) data. (b) And (c) scanning electron microscope (SEM) images at low and high magnifications, respectively.



Figure 3: Tensile curve along with a zoom in of the $\varepsilon_{\rm tot}$ portion tested at a $10^{-5}\,{\rm s}^{-1}$ strain rate.



Figure 4: Schematic representation of the built plate including one AB sample and one cylinder from which the as-machined specimens were extracted from. (b) And (c) are the geometries of the AB and AM specimens, respectively.

308 3. Results and discussion

This section describes the fatigue test results. In order to analyse these results, surface roughness, microhardness, and residual stress measurements of the multiple samples subjected to different processing conditions are first presented.

313 3.1. Surface roughness

Figure 5 shows a histogram of $R_{\rm a}$ parameter extracted from the measured 314 roughness profiles on AB, AB-SP, AM, and AM-SP samples. Similarly to 315 Kelley al. [15], the $R_{\rm a}$ value of the AB samples was about four times higher 316 in comparison with that of the AM samples. Shot peening decreased surface 317 roughness of AB samples as observed by Bagherifard *et al.* [27] and Wood *et* 318 al. [28]. However, the surface roughness reduction was lower herein with a 319 1.6 ratio reduction. Finally, shot peened increased surface roughness of AM 320 samples, as commonly observed for machined conventional materials that 321 were shot peened [20, 21]. 322

323

324 3.2. Microhardness

Figure 6 provides the measured in-depth microhardness profiles for the AB, AB-SP, AB-Ox, and AB-SP-Ox surface conditions. The horizontal dashed black line at 525 Hv is the AM microhardness baseline. Microhardness of the AB condition was higher over 100 µm subsurface with respect to the AM baseline, with a value of about 550 Hv at the surface. The finer grains



Figure 5: Histogram of the $R_{\rm a}$ values extracted from surface roughness measurement. The error bars correspond to one standard deviation of uncertainty assuming a normal distribution.

in the near-surface layer induced by the contour scanning strategy, shown on
the bottom of Figure 2a, were the cause of this increase as a consequence of
the Hall-Petch effect.

Shot peening increased microhardness at 600 Hv over 200 µm below the 333 surface as a result of the surface plastic deformations, which introduced 334 compressive residual stresses and cold-working. Surprisingly, the microhardness 335 values of both oxidation conditions, namely AB-Ox and AB-SP-Ox, upward 336 shifted through the depth. This shift over the entire microhardness profile 337 for the oxidized states suggested a change in the microstructure. The first 338 microstructural characterizations carried out by scanning electron microscopy 339 did not indicate any significant change in the microstructural state between 340



Figure 6: In-depth microhardness profiles of the AB, AB-SP, AB-Ox, and AB-SP-Ox surface conditions. The error bars correspond to one standard deviation of uncertainty assuming a normal distribution.

unoxidized and oxidized samples. These hardness increases were most likely 341 driven by modification at the scale of the nanoprecipitates. Finally, a similar 342 microhardness peak of 600 Hv was measured at the surface of the AB-SP 343 and AB-SP-Ox samples, whereas a decrease in this value was expected after 344 oxidation. Indeed at 540 °C, a thermal relaxation of the residual stresses 345 introduced by shot peening usually occured in conventional IN718 [37]. Consequently, 346 the surface residual stresses were measured on AM, AB, AB-Ox, AB-SP, 347 and AB-SP-Ox in order to quantify the thermal stress relaxation. The 348 corresponding results are presented in the next section. 349

350

351 3.3. Residual stress

Table 3 summarizes the residual stress measured at the surface of the AB. 352 AM, AB-Ox, AB-SP, and AB-SP-Ox samples. The contour hatch scanning 353 technique produced a compressive residual stress on the surface of the printed 354 samples. Indeed, the measured residual stress value of σ_{22} for the AB sample 355 was 75% higher than after machining, where the finer grains at the surface 356 were removed. Shot peening AB samples introduced compressive residual 357 stresses with values of hundreds megapascals. Finally as expected, oxidation 358 induced thermal stress relaxation, with a decrease of 31% and 55% when 359 compared to AB and AB-SP samples, respectively. This relaxation of residual 360 stresses was antagonistic with the increase of the microhardness values after 361 oxidation, as shown in Figure 6. This result suggested a modification of 362 the microstructure during oxidation that would compensate the hardness 363 decrease. Indeed, hardness measurements are complex characterizations, 364 which combines mechanical and microstructural information. As a result, 365 the decrease in hardness due to thermal relaxation of residual stresses were 366 likely compensated by fine precipitation of hardening phase in the oxidized 367 sample. 368

Finally, the level of thermal stress relaxation was in the same order of magnitude than that observed by Cammett *et al.* [37] for a conventional IN718 at a temparature of 525 °C. However, the authors thermally relaxed their shot peened samples during 10 hours, versus 1000 hours herein. Thus, a higher relaxation rate was expected in the present study. Such a dicreapancy

Table 3: Surface residual stress measurements on AB, AM, AB-Ox, AB-SP, and AB-SP-Ox samples. σ_{11} is the stress tensor component in the building direction, and σ_{22} is the non-zero stress tensor components perpendicular to the building direction, *i.e.*, in the tangential direction of the samples.

| Surface condition | σ_{11} [MPa] | σ_{22} [MPa] |
|-------------------|---------------------|---------------------|
| AB | -89.1 | -112.1 |
| AM | -81.5 | -27.3 |
| AB-Ox | -56.6 | -82.4 |
| AB-SP | -740.5 | -746.3 |
| AB-SP-Ox | -368.5 | -298.8 |

³⁷⁴ yielded to question whether the particular microstructures of additive manufactured
³⁷⁵ materials changed residual stress relaxation.

376 3.4. Fatigue results

Figure 7 provides the fatigue results of the tested AM samples. Four stress levels were applied to determine the maximum stress (σ_{max}) corresponding to a fatigue lifetime of 5×10^4 cycles to limit potential fatigue runout set at 1×10^6 cycles after shot peening. Based on these fatigue results, $\sigma_{max} =$ 650 MPa was selected for the rest of the study.

382

Figure 8 presents the fatigue lifetime data for the studied surface conditions. The y-axis indicates the baseline (BS), the oxided (Ox), the shot peened (SP), as well as the shot peened and oxided (SP-Ox) conditions. These conditions are subdivided in AB and AM conditions, which are presented in filled and unfilled symbols, respectively. Figure 8 shows a dual population



Figure 7: Fatigue lifetime results at 650 MPa for R = 0.1 at 550 °C for the AM surface condition.



Figure 8: Fatigue lifetime results at 650 MPa for R = 0.1 at 550 °C for the studied surface conditions. BS stands for baseline and corresponds to the AB and AM surface conditions

of fractures, approximately at 4×10^4 cycles and 3×10^5 cycles in average. 388 Fractography observations of the tested fatigue samples revealed surface 389 fatigue crack initiations for the samples that fractured around 4×10^4 cycles, 390 and subsurface fatigue crack initiations for the samples that fractured around 391 3×10^5 cycles. Moreover, fatigue cracks consistently initiated at lack of 392 fusion, except for the AB and AB-Ox samples that fractured around 4×10^4 cycles. 393 Both of these conditions had samples that initiated on a surface discontinuity 394 most likely induced by the L-PBF process. Figure 9 shows a few fracture 395 surfaces that were observed under SEM to support both previous conclusions. 396 Figure 9a presents a surface crack on a lack of fusion that initiated on a AM 397 sample having a fatigue lifetime of 42959 cycles. Figure 9b shows a surface 398 crack on a surface discontinuity that initiated on a AB sample having a 399 fatigue lifetime of 41 504 cycles. Figure 9c indicates a subsurface crack on 400 a lack of fusion that initiated on a AB sample having a fatigue lifetime of 401 347 614 cycles. Figure 9d points out a subsurface crack on a lack of fusion 402 that initiated on a AB-SP sample having a fatigue lifetime of 370 012 cycles. 403 Finally, Figure 9d additionally shows an irregular black outline having a 404 flower shape. This outline corresponded to the propagation period, and was 405 observed on most of the fracture surfaces. Close-up view of these flower 406 shapes revealed that porosities were the source of this irregularity in the 407 final crack front. 408

The present fractographic analysis revealed that porosities drastically undermined
the fatigue performance, since porosities were the main source of crack initiations

⁴¹¹ and reduced the crack growth periods.

412

Figure 8 also revealed that average fatigue lifetime of AB samples was higher than that of AM samples. As mentioned in the methodology section, the AB samples had a tested volume 1.8 higher than the AM samples, leading to a potential size effect on the fatigue results. However, the size effect would have led to a better fatigue lifetime for the AM samples since the tested volume was smaller.

The fact that AB samples had a better fatigue lifetime than AM samples 419 disagreed with the literature at room temperature [3, 15]. In these articles, 420 the authors listed the higher surface roughness as the prime source of fatigue 421 life reduction for the AB samples. Herein, the surface roughness of AB 422 samples was about four times higher in comparison with that of the AM 423 samples, and thus could not rationalize the observed fatigue life discrepancy. 424 The hypothesis to rationalize the fatigue life discrepancy was the beneficial 425 effect of the contour strategy. First, the contour strategy induced finer grains 426 at the surface of the AB samples, as shown on the bottom of Figure 2a. Those 427 finer grains enhanced the fatigue properties at the surface of the samples [38]. 428 Second, compressive residual stress was 75% higher on AB samples than on 429 AM samples. Finally, the layer built by the contour strategy was likely free 430 of porosities and thus protected the samples from surface crack initiation on 431 lack of fusion. This was consistent with the fractographic analyses described 432 above. Indeed, fatigue cracks of AB and AB-Ox samples never initiated at the 433

⁴³⁴ surface on a lack of fusion. The present analysis suggested that the contour
⁴³⁵ strategy had a paramount role in enhancing fatigue performance of the
⁴³⁶ AB samples. In fact, the contour strategy counterbalanced the detrimental
⁴³⁷ roughness effect on fatigue life.



(c) AB: subsurface crack initiation

(d) AB-SP: subsurface crack initiation

Figure 9: Fractographic observations of fatigue tested samples having an (a) AM, (b-c) an AB, and (d) an AB-SP surface condition.

As expected, shot peening increased fatigue life in average for AM and 439 AB surface conditions. Fatigue life improvement was 7.5 times higher after 440 shot peening, while Cammett et al. [37] observed on a conventional IN718 441 at 525 °C an improvement of only 2.6 times. Such a large improvement gap 442 was attributed to the inferior fatigue properties of the unpeened samples, 443 due to the large amount of porosities within the printed samples. In fact, 444 Cammett et al. [37] observed under an identical stress ratio at 525 °C that 445 the maximum stress reaching 4×10^4 cycles was 1500 MPa for an as-polished 446 sample, which was about 2.3 times higher than that of the printed samples. 447 In addition, Cammett et al. [37] observed that the maximum stress reaching 448 3×10^5 cycles was 1500 MPa for a peened sample, which was about 2.3 times 449 higher than that of the shot peened printed samples. Therefore, even though 450 shot peening improved fatigue life of printed samples, it did not reach fatigue 451 lifetime of shot peened conventional IN718. 452

In the present study, shot peening prevent surface crack initiation, and thus 453 push the population at 4×10^4 cycles toward 3×10^5 cycles. Shot peening 454 was previously observed to annihilate porosities near the surface [23-25]. 455 However, herein, this annihilation likely had no effect on fatigue life since 456 unpeened specimens had crack initiations either at the surface or subsurface 457 outside the shot peening affected layer. Moreover, based on the present 458 fractographic analysis, surface roughness appeared to play a minor role in 459 the fatigue process. As a consequence, compressive residual stresses were 460 most likely the main shot peening modification that prevented surface crack 461

462 initiation.

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Finally, it appears that oxidation did not affect the fatigue lifetime even 464 after shot peening where a diminution of fatigue lifetime was expected due 465 to the relaxation of compressive residual stresses. Considering, that oxidized 466 samples had identical surface roughness to the unoxidized counterpart, compressive 467 residual stress most likely led the fatigue process. It appeared that the 468 level of compressive residual stress after oxidation was sufficient to prevent 469 surface crack initiation, and thus hold back fatigue life from decreasing. As 470 a conclusion, oxidizing the samples during 41.6 days at 550 °C dit not impact 471 the fatigue life under the studied stress conditions. 472

473 4. Conclusion

Additive manufactured IN718 samples were fatigue tested at 550 °C, with $\sigma_{\rm max} = 650$ MPa, and R = 0.1. As-built, as-machined, shot peened, and oxidized surface conditions were studied. The following conclusions were obtained:

- As-built samples had better fatigue lifetime than that of as-machined
 samples. The contour strategy used to print the samples was considered
 responsible of this beneficial effect.
- Shot peening strengthened the beneficial effect of the contour strategy
 on fatigue life.

• Oxidizing the samples during 41.6 days at 550 °C did not impact the fatigue lifetime.

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