Low- and high-cycle fatigue resistance of Ti-6Al-4V ELI additively manufactured via Selective Laser Melting: Mean stress and defect sensitivity

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Abstract

Selective laser melting (SLM) is a net-shape AM technology to produce metal parts also for load-carrying applications. The present work is aimed at investigating the fatigue performance of the biomedical titanium Grade 23 (aka Ti-6Al-4V ELI) AMed via SLM. Low and high cycle fatigue tests are carried out on samples that received a low temperature stress-relief treatment. In addition, the effect of selected post processing treatment on the high cycle fatigue response is assessed. Material characterization is complemented with residual stress and microhardness measurements, computed tomography scans, metallographic and fractographic inspections. These experimental analyses served to elaborate an interpretative model accounting for the modifications produced by the post-processing treatments. The results denote the important role exerted by mean and residual stresses as well as defects on the fatigue performance. The relatively low fatigue strength of SLM manufactured parts indicates that further developments in this fabrication route are still necessary to make their mechanical properties competitive with those of traditionally processed components.

Keywords

Selective laser melting; titanium; low and high cycle fatigue; defects; computed tomography

Nomenclature

Symbols

AM	additive manufacturing
area	area obtained by projecting a defect or a crack onto the plane *x*-*y* perpendicular to the maximum tensile stress
b	fatigue strength exponent (Eq. (1a))
c	fatigue ductility exponent (Eq. (1b))
c_1, c_2, *m*	parameters of Eq. (4) used to fit high cycle fatigue data
CT	computed tomography
E	Young's modulus
F	cumulative probability
H'	Ramberg-Osgood coefficient (Eq. (3))
HCF	high cycle fatigue
HIP	hot isostatic pressing
HV	Vickers hardness
LEVD	largest extreme value distribution
LCF	low cycle fatigue
LOF	lack of fusion
n'	Ramberg-Osgood exponent (Eq. (3))
N_f	number of cycles to failure
R	load (stress) ratio
S^2	estimated regression variance (Eq. (5))
SEM	scanning electron microscope
SLM	selective laser melting

SWT	Smith-Watson-Topper model [43]
y	reduced variate of LEVD
V	defect volume
α, β, γ	material parameters of Eq. (7)
ε	axial strain
ε'_f	fatigue ductility coefficient (Eq. (1b))
σ	axial stress
σ'_f	fatigue strength coefficient (Eq. (1a))
σ_Y	yield stress
σ_U	tensile strength
ϕ	non-dimensional defect shape factor (Eq. (6))

Subscripts

a	amplitude
el	elastic
m	mean
pl	plastic

1. Introduction

Additive Manufacturing (AM), sometimes colloquially termed 3D-printing, comprises net-shape production technologies that build a solid object from the sequential superposition of layers representing the cross-sections obtained by virtually slicing the 3D model of the component. Nowadays, AM is becoming a key enabling technology for direct fabrication of functional or structural end-use products and is already revolutionising not only the way we produce, but also the design guidelines. Advantages offered by AM over conventional subtractive or formative techniques stem from broader design freedoms that allow geometries of virtually any complexity to be manufactured with minimal tooling, rapid delivery times, and low material waste [1,2].

Since 1990, several AM technologies have been developed to sinter metallic powders. They can be distinguished regarding the way the layers of material are deposited and consolidated [3,4]. In powder bed fusion processes, the powder is spread to a controlled thickness (typically on the order of 0.1 mm) over the build platform or the previously built layers. After powder consolidation, the build platform is lowered and a new layer is spread. The process repeats until the entire model is created. Different heat sources are used to sinter or fuse the powder. For instance, a laser or an electron beam is adopted in Selective Laser Melting (SLM)/Selective Laser Sintering (SLS)/Direct Metal Laser Sintering (DMLS) or Electron Beam Melting (EBM), respectively [5,6]. The former operate in an inert environment, the latter in vacuum. In all cases, the heat input is intense and highly localized so that the process parameters must be carefully tuned, especially in terms of scan speed, pattern and energy density [7].

SLM is well suited to additively manufacture small-to-medium amounts of parts with moderate-to-high surface finish [2]. Titanium and its alloys are frequently manufactured via SLM, mainly in the biomedical, aerospace and energy context, to obtain components of high-added value that justifies the high production costs of this fabrication route. Meaningful examples thereof are turbine blades with internal conformal cooling channels [8] and customized medical implants even with cellular or porous structure for improved osseointegration [9,10]. The geometrical complexity of these components, often inaccessible to conventional subtractive/formative manufacturing techniques, and the poor machinability of Ti alloys, linked to the high energy required to remove/deform the material, make AM in general and SLM in particular the only viable solution from a technical or economical standpoint.

Among Ti alloys, Ti-6Al-4V, hereinafter abbreviated in Ti64, is the most important, covering about 60% of the entire world market [11]. This two-phase α+β Ti alloy combines superior fatigue strength-to-weight ratio, high operating temperatures, excellent corrosion resistance and biocompatibility [12]. A specific advantage resides in the wide range of microstructural options that can be obtained by thermo-mechanical processing and heat treatment, allowing one to balance the fatigue properties with other design limiting properties (strength, stiffness, fracture toughness, etc.). Unlike other metallic materials for structural applications, such as steels and Al alloys, wrought Ti alloys display clean microstructures without the presence of hard inclusions, which impact detrimentally on ductility and fatigue resistance. The most important microstructural parameter determining the mechanical properties of α+β Ti alloys is the α colony size, which

controls the maximum dislocation slip length and, as a consequence, its mechanical properties [13]. Basically, α+β-processed fine-grained (equiaxed or bimodal) compared to β-annealed fully lamellar microstructures show superior fatigue resistance as a consequence of the smaller α colony size, in general at the cost of a lower crack growth resistance, which is instead a peculiar characteristic of the latter coarse microstructures conferred by extrinsic crack-tip shielding mechanisms [14].

When Ti-64 is additively manufactured (AMed) via SLM, the mechanical properties are less dictated by the aforementioned relationships. In fact, the microstructure resulting from the very high cooling rate from the β-field is in general composed of an acicular α' hcp phase, a metastable martensitic phase displayed by the α+β Ti-alloys [15-17]. It is characterized by low ductility, hence not adequate for most structural applications, especially because the mechanical properties are further degraded by the presence of defects and internal stresses produced by the high thermal gradients affecting the SLM process [18]. Typical flaws are pores produced by initial powder contamination, evaporation or local voids after powder-layer deposition [16,19] and lack of fusion (LOF) defects. These latter are caused by insufficient energy density resulting in incomplete powder bed consolidation and generally assume the form of irregularly shaped cavities that may entrap unmelted powder particles [20,21].

The combination of low ductility, defectiveness and residual stresses can be very critical to the fatigue strength of SLM products. Therefore, the fatigue characterization of SLM manufactured Ti-64 has attracted in recent times the attention of the research community [20-27]. Kasperovich and Hausmann [22] carried out a systematic investigation of the effect of SLM process parameters on the fatigue resistance of Ti-64, observing that the detrimental presence of porosity can be minimized adopting moderate laser scanning velocity and large spot size. A further decrease in porosity was achieved via hot isostatic pressing (HIP). Liu et al. [20] investigated the onset and morphology of LOF defects in SLM manufactured Ti-64, noting that their effect on the fatigue resistance is more detrimental in the vertically than the horizontally built samples. Nicoletto [23] made similar observations for DMLS fabricated Ti-64. The pioneering works of Leuders et al. [24,25] were among the first to study post-process treatments with the aim of improving the fatigue resistance of SLM manufactured Ti-64. They found that heat treatments conducted either below or above the β-transus improve ductility and hence the fatigue strength and that HIP brings porosity below the minimum level detectable by X-ray computed tomography (CT), increasing the fatigue strength to values comparable

to conventionally processed Ti-64. Edwards and Ramulu [26] carried out fatigue tests on SLM fabricated Ti-64 in the as-built condition, observing that the fatigue strength is 75% lower than the corresponding wrought material due to poor surface finish, porosity and surface tensile residual stresses. Greitemeier et al. [27] found that the fatigue strength of Ti-64 produced by DMLS is mainly controlled by surface roughness and internal defects. The role of microstructure becomes important only if the latter effects are eliminated. In this case, fine microstructures are preferable to the coarse lamellar ones typically obtained after HIP. Günther et al. [28] explored the very-high-cycle-fatigue resistance of additively manufactured Ti-64 using an ultrasonic testing machine. They distinguished two failure modes, i.e. surface fatigue crack initiation, typical of HIPed samples and localized at α-phase clusters, and internal fatigue crack initiation, occurring in as-built and heat-treated samples near pores and LOF defects. HIP greatly improves fatigue resistance; further increments are possible only by decreasing the α -colony size, as for wrought components. A systematic analysis of the fracture surfaces was undertaken with the aim of interpreting the fatigue results by the light of the $\sqrt{area}$ model originally developed by Murakami [29] for steels and cast irons, whose fatigue behaviour is mainly dictated by inclusions [30] and shrinkage porosity [31], respectively. The same approach was adopted by Beretta and Romano [32], who were able to rationalize the large variety in fatigue data published in the open literature by correlating the data to the defect characteristic size $\sqrt{area}$, thus confirming the applicability of this method to AMed Ti components. Li et al. [33] presented a comprehensive review of published fatigue data of AMed Ti-64 outlining the lower fatigue performance with respect to conventionally processed components and the necessity for post-processing treatments. To conclude, they give illuminating indications for further investigations, highlighting the need for (i) systematically investigating the effect of the stress ratio *R*, further motivated by the anomalous mean stress sensitivity found by Oberwinkler [34] for wrought Ti-64; (ii) designing specimens able to reduce stress concentration and representative of the actual component microstructure and defectiveness; (iii) exploring long fatigue lives, beyond 10^7 cycles usually taken as run-out tests; (iv) strain-controlled testing to explore cyclic stress-strain and low-cycle-fatigue (LCF) behaviour, which is important especially in the presence of stress raisers.

Taking inspiration from the suggestions for future research outlined in [33], the present paper is aimed at enriching the understanding of the fatigue properties of the biomedical Ti Grade 23, aka Ti-6Al-4V ELI, AMed via SLM. Specific points of novelty with respect to the existing technical literature are: (i) the fatigue

tests are conducted up to the very-high-cycle-fatigue regime (up to 5×10^7 cycles), (ii) employing specimens intentionally provided with uniform gage length directly manufactured by SLM (without any post-process subtractive machining) to emphasize the effect of defectiveness located in particular on the outer layers; (iii) the effect of three stress-ratios on the high-cycle fatigue response is experimentally investigated; (iv) strain-controlled LCF tests are carried out to gain information about cyclic strain hardening, cyclic stress-strain curve and Basquin/Coffin-Manson equation; (v) several experimental techniques, such as microhardness, X-ray diffraction, X-ray computed tomography scans, metallographic and fractographic analyses, are combined together to collect the information necessary to devise an interpretative model of the fatigue behaviour of the material subjected to different post-process treatments.

2. Experimental material and procedures

2.1 Specimen preparation

The experimentation is carried out on cylindrical samples provided with a smooth transition from the uniform gage section in the centre to the grip section at the ends, as shown in Fig. 1. In this way, (i) the stress concentration factor at the transition is very low (about 1.02), and (ii) a significant amount of material is critically stressed, so that the impact of microstructure and defectiveness on the fatigue behaviour can be captured more effectively compared to common hourglass specimens.

The samples are additively manufactured by SLM along the longitudinal *z* direction, using a 3D Systems ProX 300 printer and an atomized powder of the biomedical Titanium Grade 23, also indicated as Ti-6Al-4V ELI. The powder size is very fine (average diameter of about 9 μm) so as to ensure good surface finish. Further details can be found in [35]. The process parameters (laser power, hatching spacing, scan speed) were optimized by the manufacturer and are confidential.

The resulting microstructure, shown in Fig. 2a, is composed of a very fine acicular α' martensite produced by the high cooling rate of the SLM process. In order to eliminate internal stresses and increase material ductility, all the samples are heat-treated after SLM at 670 °C for 5 h in a protective Ar-atmosphere. Surface finish is not altered by post-processing treatments. Specimens in this condition will be referred to as "as-built".

Part of the samples is subject to a HIP treatment with the aim of reducing as much as possible the porosity on the entire sample cross-section. During this treatment, high temperature and high pressure are concomitantly applied to induce plastic flow and consequent nearly full densification of the part. Special care must be taken in selecting the proper HIP temperature in order to optimize the resulting microstructure. For this purpose, as-built samples are heat-treated at different temperatures using a Baehr DIL 805 dilatometer. Specifically, samples are heated with a rate of 5°C/min, maintained at constant temperature for 2 h and eventually cooled down to room temperature with the same rate. Figures 2b, c, and d illustrate the microstructure obtained setting the isothermal stage at 900°C, 920°C and 950°C, respectively. At 900°C (Fig. 2b), α-platelets start forming, preferentially at the grain boundaries, from the metastable α' phase. At 920°C (Fig. 2c), the microstructure is composed of α-platelets interspersed in the β matrix, a further increment in temperature up to 950°C (Fig. 2d) makes the platelets coarser. This analysis indicates how the evolution of the martensitic microstructure is sensitive to the annealing temperature and advices against HIP at excessive temperature, especially above the β-transus (about 980°C for Ti-64), as it can result in undesired overgrowth of the α-platelets. On the base of these outcomes, the HIP treatment is conducted at 920°C, 1000 bar of pressure for 2 h in Ar protective atmosphere. The resulting microstructure, shown in Fig. 2e, is very similar to that depicted in Fig. 2c and obtained after annealing at the same temperature. Prior to HIP, the samples receive a tribofinishing treatment to improve the surface finish.

To assess the influence of the sole microstructural changes imparted by HIP on the fatigue behaviour, some of the as-built samples received the same heat treatment as HIP yet without pressure application. For this purpose, the as-built samples are heat-treated at 920°C for 2 h using a furnace operating in vacuum to minimize O and N contamination and eventually furnace-cooled down to room temperature. Specimens in this condition will be referred to as "annealed".

An electropolishing treatment, specifically developed in [36] for biocompatible applications, is applied to part of the as-built specimens in order to evaluate the effect of the surface morphology on the fatigue response. It employs an alcoholic solution of aluminium chloride and zinc chloride. Further details are given in [35].

Finally, some as-built samples are shot peened to an Almen intensity of 6.4A and 200% coverage using ceramic (ZrO_2 and SiO_2) beads of 300 – 425 μm diameter; details of the experimental setup are presented in

[35]. This treatment is compliant with biocompatibility requirements and its intensity was intentionally calibrated in order not to increase the surface roughness of the samples.

2.2 Mechanical tests

Monotonic tensile tests (initial strain rate of 1×10^{-4} s^{-1}) are performed at room temperature (25°C, 60% R.H.) on cylindrical specimens in as-built, annealed and HIPed conditions on a servo-hydraulic universal testing machine INSTRON 1343 (Instron, Nordwood, MA, USA), equipped with hydraulic grips, a load cell of 100kN (nonlinearity ± 0.1% of R.O.) and an axial extensometer (10 mm gauge length, nonlinearity ± 0.15% of R.O.). The yield strength is determined as the 0.2% offset yield stress.

To evaluate the cyclic stress-strain and the LCF behaviour, strain controlled fatigue tests are performed on as-built specimens according to the standard ASTM E606. Specifically, each sample is tested at constant strain amplitude until final failure using a constant strain rate of 1×10^{-2} s^{-1}. Fully reversed strain amplitudes (strain ratio R_ε = - 1) are applied at 5 strain amplitudes comprised in the range [0.003,0.010]. Overall 13 specimens are used, thereof two specimens are used for the largest and the lowest strain amplitude, and three samples are used for the intermediate ones. Using only two samples at the highest strain level is acceptable as the dispersion in the fatigue lives is very small. Using two samples at the lowest strain amplitude is acceptable as well since the Basquin/Coffin-Manson equation agrees well with the outcomes of the load controlled HCF tests at similar strain amplitudes (see Section 3.4).

The LCF data are elaborated by dividing the total strain amplitude of the stabilised hysteresis loops into its elastic and plastic components:

$$\varepsilon_a = \varepsilon_{a,el} + \varepsilon_{a,pl} \tag{1}$$

which are then separately fitted according to the Basquin and Coffin-Manson equations, respectively:

$$\varepsilon_{a,el} = \frac{\sigma'_f}{E}\left(2N_f\right)^b \tag{1a}$$

$$\varepsilon_{a,pl} = \varepsilon'_f\left(2N_f\right)^c \tag{1b}$$

The elastic and plastic part of the strain amplitude is computed from stabilized half-life stress-strain hysteresis loops as:

$$\varepsilon_{a,el} = \frac{\sigma_a}{E} \tag{2a}$$

$$\varepsilon_{a,pl} = \varepsilon_a - \varepsilon_{a,el} \tag{2b}$$

where E is the Young's modulus. The half-life hysteresis loops of the LCF tests have been used to determine the cyclic stress-strain curve of the material. The cyclic and monotonic stress-strain curves have been fitted using the Ramberg-Osgood equation:

$$\varepsilon = \frac{\sigma}{E} + \left(\frac{\sigma}{H'} \right)^{1/n'} \tag{3}$$

To evaluate the high-cycle-fatigue (HCF) behaviour, axial fatigue tests are carried out in laboratory environment using a resonant testing machine Rumul Mikrotron 20kN (Russenberger Prüfmaschinen, AG, Neuhausen am Rheinfall, Switzerland) operating at a nominal frequency of 150 Hz under load control. Alternating (zero mean stress, stress ratio $R = -1$) fatigue tests are conducted on samples in all the conditions illustrated in the previous section. To investigate the mean stress sensitivity, additional fatigue tests are performed on as-built samples at stress ratios $R = -3$ and $R = 0.1$. Tests are carried out at different stress amplitudes to explore fatigue lives comprised between 10^4 and 5×10^7 cycles. Overall, 80 coupons are tested; the S-N curve for each experimental condition is obtained from 9 to 15 samples. Run-out tests are terminated at 5×10^7 cycles when no facture takes place, a fatigue life regime not explored in the researches reviewed by Li et al. [33] and surpassed only by Günther et al. [28] using an ultrasonic testing apparatus.

Two types of fatigue curves are obtained, i.e. without and with knee located around 10^6 fatigue cycles. The former fatigue curves are represented by the common S-N Basquin equation:

$$\sigma_a = c_1 \times N_f^{-\frac{1}{c_2}} \tag{4a}$$

The latter fatigue curves are represented by a S-N curve with an asymptotic behaviour expressed by:

$$\sigma_a = c_1 + \frac{c_2}{N_f^m} \tag{4b}$$

The scatter of the fatigue data is assessed by computing the estimated regression variance assumed to be uniform for the whole fatigue life range and expressed by:

$$S^2 = \frac{\sum_{i=1}^{n}\left(\sigma_{a,i} - \hat{\sigma}_{a,i}\right)^2}{n-p} \tag{5}$$

where $\sigma_{a,i}$ is the i-th fatigue amplitude data point, $\hat{\sigma}_{a,i}$ is its estimator, n is the number of data elements, and p is the number of parameters in the regression; p = 2 and 3 for Eq. (4a) and (4b), respectively.

2.3 Material characterization

The various material conditions are experimentally characterized through surface roughness, microhardness, X-Ray Diffraction (XRD) residual stress. Details about the experimental procedures are given in [35]. In the present work, additional residual stress measurements are undertaken on a shot peened sample that has been previously tested in the high-cycle fatigue regime (σ_a=350 MPa, N_f = 32×10^6 cycles) in order to investigate the effect of fatigue loading on the stability of the residual stress field. Specifically, measurements are performed after fatigue failure in a region far enough from the fracture surface (about 2 mm) so that the material rupture is supposed not to have altered the residual stress field.

A specific aim of the present paper is to investigate the effect of defectiveness on the fatigue behaviour of SLM manufactured components. For this purpose, micro computed tomography (CT) is used, as it is an effective technique to evaluate internal defects of additively manufactured parts, providing complete information on pores' spatial distribution, size and morphology [37]. CT scans are undertaken on a volume of about 40 mm^3 located in the central uniform gage section of samples in the as-built, HIPed and shot-peened conditions. To achieve a direct comparison between the as-built and the peened condition, the same sample is scanned prior to and after the peening treatment. A metrological CT system Nikon X-Tek MCT225 (Nikon Instech Co. Ltd., Tokyo, Japan) is employed with micro-focus X-ray tube (minimum focal spot size equal to 3 μm), 16bit flat panel detector with 2000×2000 pixels and controlled cabinet temperature at (20 ± 1) °C. Each sample is scanned with voxel size equal to 2.8 μm and using the same scanning parameters (see Table 1) for an accurate comparability of results. The low scanning power allowed keeping the focal spot size at a minimum, achieving the best resolution. Porosity analyses are conducted on the CT reconstructed three-dimensional models using the software VGStudio MAX 3.0 (Volume Graphics GmbH, Germany). To enhance the accuracy of porosity analyses, the threshold value for such analyses is set by averaging the grey

values of material's voxels and grey values of voxels inside pores, according to the procedure proposed in [38]. In particular, the volume V, the area *area* projected on the transversal *x*-*y* plane (as schematically shown in Fig. 3), and the minimum distance from the outer surface are calculated for each detected pore. The deviation of the projected area from that of an ideally spherical pore of same volume V is estimated according to the following non-dimensional parameter:

$$\phi = \frac{area}{\pi^{1/3}\left(\frac{3}{4}V\right)^{2/3}} \tag{6}$$

which takes value 1 for a spherical pore and becomes larger than 1 for pores stretched along the *x*-*y* plane.

CT scans are complemented by 2D metallographic porosity measurements. For this purpose, transversal sections are extracted from the samples orthogonally to the longitudinal axis *z*. After polishing, optical micrographs are taken at 100x magnitude and stitched together to reconstruct the entire cross-section. The pores are measured in size and location using the software ImageJ®. To get a robust statistical analysis of the porosity, five measures are replicated for each condition. In addition, metallographic analyses are undertaken after CT scans on the shot peened and HIPed samples to get information from the two techniques on the same experimental specimens.

3. Experimental results

3.1 Monotonic tensile properties

The results of the monotonic tensile tests are listed in Table 2. Even though, due to the high cost for specimen production, only one sample is used per each microstructural condition, it is reasonable to affirm that the static properties of the material are significantly influenced by the microstructure. In particular, the as-built condition characterized by a stress-relieved martensitic microstructure exhibits the highest yield stress and tensile strength as well as the lowest total elongation, however compliant with the minimum value prescribed for biomedical devices according by the standard ASTM F2924 – 14. The annealed and the HIPed conditions display similar tensile properties, as they received the same heat treatment in the $\alpha+\beta$ phase field. Apparently, the effect of the pressure application on the static properties and the resulting reduction in porosity seems to be marginal, as already observed by Leuders et al. [24].

3.2 Surface morphology, microhardness and residual stress profiles

The results of surface roughness are listed in Table 3. The as-built condition is affected by the largest roughness, even though a Ra value of 7 μm can be regarded as fairly low in comparison with values reported in the literature for SLM manufactured components [26]. This has been achieved thanks to the very fine powder used in the present SLM process. Electropolishing dramatically reduces the roughness down to the lowest value explored in this paper. The HIPed variant shows a roughness lower than the as-built condition owing to the tribofinishing treatment applied prior to HIP. Finally, shot peening is able to bring the surface roughness below that of the as-built condition. More analyses about the surface morphology can be found in [35].

Figure 4a compares the in-depth microhardness profiles of shot-peened and HIPed specimens. Since the microhardness profiles measured on the as-built and annealed conditions do not show any clear trend with respect to the depth below the surface, the corresponding hardness measurements are plotted in Fig. 4a as mean value and standard deviation taken constant throughout the depth. It can be noted that the as-built variant displays an average microhardness of about 380 HV with a standard deviation of 10 HV. Transforming the martensitic α' into α+β processed microstructure decreases the microhardness by about 10%, viz. a relative decrement comparable with that in yield stress, as shown in Table 1. The strain hardening introduced by the shot-peening process increases the hardness of the surface layers, over a depth below the surface varying between 0.15 and 0.30 mm, respectively. The HIPed condition displays lower microhardness values due to the softer α+β processed microstructure. The large surface microhardness can be explained by the intense plastic deformation produced by the thermo-mechanical treatment on the surface layers. It is interesting to notice that the microhardness profiles of both shot-peened and HIPed variants approach the microhardness of the parent microstructure below the strain-hardened surface layer.

Figure 4b illustrates the in-depth residual stress profiles measured in the as-built, shot peened and HIPed variants. Since electropolishing and annealing do not significantly alter the residual stress field, the corresponding profiles are not shown in Fig. 4b for the sake of clarity. It can be noted that the long stress-relief treatment is able to nearly eliminate SLM induced internal stresses from the as-built condition. As expected, shot peening (here refer to the dataset indicated as "initial" in Fig. 4b) introduces the highest compressive residual stresses, with a characteristic sub-superficial peak of -700 MPa located about 30 μm

below the surface. It is interesting to notice that the strain hardening produced by HIP on the surface layers, as attested by the microhardness profile of Fig. 4a, introduces moderate surface compressive residual stresses of about -400 MPa.

3.3 Porosity

Porosity is computed from metallographic inspections and CT scans in four concentric annular areas depicted in Fig. 5a. The corresponding results are plotted in Fig. 5b. It can be noticed that the porosity values estimated by the two very different experimental techniques are in quite good agreement. In fact, the CT measurements (open symbols) lie within the error band of the metallographic analyses (closed symbols). In general, the CT scans tend to systematically estimate lower porosity than the metallographic inspections. This discrepancy can be attributed to several factors discussed in [37] and to the fact that the present 2D analyses consider only 5 cross-sections, while the CT scans analyse the entire volume of the specimen. Anyway, the two techniques depict the same trends in terms of spatial distribution of the pores inside the sample and effect of post-process treatments on the defectiveness. In particular, the as-built condition exhibits the highest porosity of about 0.35% in the outermost area 4, while it declines significantly moving towards the sample interior. The high porosity of the outer layer is probably produced during the initial contour scanning, which is conducted at a higher scan speed than that adopted in the rest of the cross-section. The effect of HIP on the defectiveness is remarkable; in fact, porosity lies below 0.05% throughout the sample. Shot peening instead has no significant effect on the porosity, as attested by both CT and metallographic analyses. This contradicts the outcomes of [35], where shot peening was found to reduce the porosity of the outer layer. This discrepancy can be explained by the fact that (i) different samples were tested in [35], so the initial porosity of the peened sample could be different from that of the sample in the as-built condition and (ii) that an insufficient number of metallographic sections was analysed in [35], so that large pores could have been overlooked. In conclusion, Figure 5c illustrates the value of the average non-dimensional shape coefficient ϕ. It can be noted that shot peening and HIP reduces ϕ, viz. the tendency of the pores to be more elongated along the transversal plane *x*-*y*. As discussed in more detail in the following, shot peening is more effective in changing the shape of the pores rather than in reducing their volume.

3.4 Cyclic and low-cycle fatigue properties

The evolution of the cyclic stress amplitude is shown in Fig. 6a as a function of the number of cycles for all the tested strain amplitudes. It can be noted that the as-built material exhibits severe strain softening at the highest tested strain amplitudes. A similar behaviour is displayed by steels with martensitic and bainitic microstructures [30,39]. At the lowest strain amplitudes, the stress amplitude remains fairly stable throughout the fatigue life.

Stabilized half-life stress-strain hysteresis loops are shown in Fig. 6b. At low strain amplitudes, the deformation is essentially elastic. Anyway, even at the highest strain amplitudes, the entity of the plastic deformation remains small as a consequence of the very high yield stress displayed by the as-built material. The elastic part of the strain amplitude is estimated according to Eq. (2a), where E is taken as the Young's modulus estimated from monotonic tensile tests. It is worth noticing that this value is very close to the slope of the elastic unloading ramp of the hysteresis loops prior to inverse plastic deformation. This behaviour is very different from that displayed by steels in which the cyclic (pseudo-) Young's modulus decreases with increasing strain amplitude [30,40].

The results of the LCF tests are summarized in Fig. 6c. The best-fit coefficients of the Basquin and Coffin-Manson equations (Eq. (1)) used to interpolate the experimental data are summarized in Table 4. The half-life hysteresis loops of the LCF tests have been used to determine the cyclic stress-strain curve of the material. The cyclic and monotonic stress-strain curves have been fitted using Eq. (3). The best-fit parameters are listed in Table 5. Figure 6d compares the cyclic stress-strain curve with the monotonic curve. It can be noted that the LCF cyclic curve lies below the monotonic curve; hence the material undergoes moderate cyclic softening, even though extrapolating the cyclic curve at strain levels above 0.01 results in a steeper rising behaviour than the monotonic curve.

3.5 High-cycle fatigue properties

Figure 7a compares the results of the fully reversed (R = -1) axial fatigue tests carried out on all the material variants, while Figure 7b illustrates the results of the axial fatigue tests undertaken on the as-built condition at three different stress-ratios. Fitting curves corresponding to 50% failure probability, expressed by Eqs. (4),

are also plotted in Figs. 7. The best-fit parameters are listed in Table 6, which reports also the standard deviation S calculated as the root square of the estimated regression variance S^2.

The fatigue strength of the as-built condition is relatively low, the fully reversed fatigue endurance at 50 million cycles is about 20% of the material tensile strength and is about half of the fatigue limit reported in the literature for conventionally processed Ti-64 [11]. The scatter in fatigue data is also remarkable, being about 10% of the high-cycle fatigue resistance. The results of the load controlled fully reversed HCF tests are reported also in Fig. 6c along with those of the strain-controlled LCF experiments. It can be noted that Eq. (1) yields fatigue strength predictions comparable to the HCF results not only at strain/stress level common to both type of fatigue tests but also at longer fatigue lifetimes. This suggests the fact that similar fatigue damage mechanisms control both LCF and HCF behaviour of the as-built material, apparently due to its limited plastic capability.

Annealing in the α+β phase field produces even a slight decrement in fatigue strength, apparently due to the softer microstructure (see Fig. 4a). Conversely, electropolishing and, to a much greater extent, shot peening and HIP produce an increment in fatigue strength and a slight reduction in scatter. All the SN curves display an asymptotic behaviour with increasing fatigue lifetime, well represented by Eq. (4b), with the only exception of the shot peened variant, which shows a steadily declining SN curve according to Basquin Eq. (4a) and occurrence of failure throughout the explored fatigue lifetime. A similar behaviour has been observed in shot-peened samples of Al-7075-T651 [41]. This behaviour will be further analysed in the following and seems not be linked to residual stress relaxation. Indeed, as shown by Fig. 4b, the stabilised residual stress field in the peened sample fatigued for about 20 million cycles is similar to the initial one, with only a partial relaxation of the surface residual stress and sub-superficial peak along the axial direction. Apparently, the high monotonic and cyclic yield strength displayed by the material limits residual stress relaxation along the loading axis induced by plastic flow during the compressive part of the fatigue load cycle [42].

The fatigue curves depicted in Fig. 7b indicate the marked mean stress sensitivity of the as-built material, highlighting the notable detrimental (beneficial) effect exerted by tensile (compressive) mean stresses on the fatigue strength. Figure 7c displays the Haigh diagram collecting the outcomes of the HCF and monotonic tensile test campaigns. It can be noted that the mean stress sensitivity of the material is not captured by

classical approaches based on Goodman and Gerber equations. Conversely, the FKM [43] and, to an even higher extent, the Smith-Watson-Topper methods [44] are in good agreement with the experimental results, thus indicating the important influence of the maximum stress on the fatigue behaviour of the material, as also observed in conventionally processed Ti-64 [34].

3.6 Fractographic analysis

SEM micrographs of fracture surfaces typically observed in the fatigued specimens are illustrated in Fig. 8. It can be noted that in all the material variants, with partial exception of the HIPed condition, the fatigue crack initiation site is located in the proximity of a defect, indicated by a red arrow and circumscribed by a dashed line. Figure 8a depicts the fracture surface of an as-built specimen, where a defect located about 100 μm below the outer surface promoted the crack initiation. It is composed of a pore of nearly spherical shape and a LOF defect with flattened morphology. Other pores, located deeper below the surface, can be observed in the neighbourhood of this critical pore. Similar appearance is presented by the fracture surface of the annealed condition shown in Fig. 8b. Note the LOF defect, located nearly 300 μm below the facture surface, responsible for the fatigue crack initiation. The fracture surface of an electropolished sample is shown in Fig. 8c. It can be noted that the material removal produced by the surface treatment has exposed to the outer surface a pore that acted as crack initiator. Figure 8d depicts the fracture surface typically observed in shot-peened samples. Unlike the previous conditions, the pore promoting crack nucleation lies well below the surface, at a depth of about 200 μm, viz. beneath the surface layer affected by compressive residual stresses and work hardening. Figure 8e and f illustrate the fracture surfaces of HIPed samples tested at low and high stress amplitudes, respectively. It can be noted that in the former case, namely at long fatigue life, crack initiation occurred near a sub-superficial pore of very small size (mean diameter 20 μm), while in the latter one, viz. at short fatigue life, the crack nucleated from the surface in the vicinity of a shallow crater.

4. An interpretative model of mean-stress and porosity sensitivity

From the fractographic inspections discussed in Section 3.6, it is clear that the defectiveness plays a crucial role on the fatigue behaviour of SLM manufactured parts. To shed further light on this matter, the results of the CT scans presented in Sections 2.3 and 3.3 are elaborated with the aim of estimating the expected

maximum defect size $\sqrt{area}_{\max}$ according to the statistics of largest extreme value distribution (LEVD) using the Maximum Likelihood Method [29,45]. Figure 9a shows the statistical distribution of defect sizes detected in the as-built, shot peened, and HIPed variants. Note the dramatic reduction in the maximum expected defect size induced by HIP. Shot peening as well reduces it slightly, apparently due to the defect shape modifications discussed in Section 3.3. It is also interesting to notice that the size of the defect that caused crack initiation in the as-built and annealed specimens shown in Fig. 8a and b corresponds to a cumulative probability *F* of 98.5% and 99.9%, respectively. This suggests the idea that the maximum defect controls the fatigue behaviour of this material; in the following the maximum expected defect size is conventionally set equal to that having a cumulative probability *F* of 99.9%. Figure 9b shows how the defects of largest size are distributed inside the samples. For this purpose, the statistic of LEVD is applied to the population of defects located at a certain depth below the outer surface. It can be noted that the largest defects are located in the outermost layers, viz. 150 μm and 250 μm below the surface in the HIPed and in the as-built and shot-peened specimens, respectively. Shot peening is able to reduce the size of the largest defects located in the outer 0.5 mm thick layer.

In light of these observations, we tried to interpret the fatigue results of all the material variants investigated so far using the well-known Murakami $\sqrt{area}$ model [28,44], expressed by:

$$\sigma_W = \frac{F_{Loc} F_{HV}}{\left(\sqrt{area}_{\max}\right)^{1/6}} \left(\frac{1-R}{2}\right)^{\gamma}$$

$$F_{HV} = \alpha \cdot HV + \beta$$

$$F_{Loc} = \begin{cases} 1.43 & \text{Surface defect} \\ 1.41 & \text{Near surface defect} \\ 1.56 & \text{Internal defect} \end{cases} \tag{7}$$

where σ_W is the predicted fatigue resistance (expressed in MPa), *R* is the stress ratio, F_{Loc} is the location factor, F_{HV} is a parameter depending upon the Vickers hardness *HV*, $\sqrt{area}_{\max}$ is the maximum expected defect size (expressed in μm). Murakami proposed F_{HV} to be a linear function of *HV* and found this relation to be valid for a wide range of steels and cast irons. In the present work, the material constants α, β, and γ are determined by least-square fitting the fatigue endurance at 5×10^7 cycles of the as-built and annealed conditions at different stress ratios using hardness and maximum expected defect size indicated in Figs. 4a

and 9a, respectively. In this way, the characteristics of the AMed material are represented in a more faithful fashion compared to the original Murakami model. Best-fit parameters and comparison between experimental data and Eq. (7) are shown in Fig. 10. The same model is then used to interpret the fatigue behaviour of the remaining material variants. For this purpose, the in-depth microhardness (Fig. 4a), residual stress (Fig. 4b) and maximum defect size (Fig. 9b) profiles are incorporated into Eq. (7) with the aim of estimating the local fatigue strength of the treated sample. Specifically, residual stresses are treated as mean stresses and incorporated into the expression of the stress ratio R.

Figure 11a compares predicted and experimental values of the fatigue strength of the as-built and electropolished conditions. The weakest point of the as-built (and similarly of the annealed) variant is located 0.15÷0.25 mm below the surface, in good agreement with the fractographic analyses shown in Figs. 8a and b. If the outer 0.5 mm thick material layer is removed by electropolishing, pores of smaller yet remarkable size are exposed to the outer surface. They act as crack initiators, as also confirmed by the SEM micrograph shown in Fig. 8c. Accordingly, the minimum fatigue strength is achieved on the electropolished surface and this predicted value is in very close agreement with the experimental one, so the limited increment in fatigue strength produced by electropolishing can be explained by the persistent presence of large pores.

Figure 11b compares predicted and experimental values of the fatigue strength of the shot peened and HIPed conditions. It can be noted that the work hardening and compressive residual stresses introduced by shot peening suppress the detrimental effect of the largest pores in the outer surface layer. In fact, the predicted fatigue strength of this layer is much higher than the experimental value. On the other hand, the minimum fatigue strength is achieved just below the treated surface, viz. at a depth of 0.15÷0.20 mm. Pores in this location become preferential crack initiation sites, as attested by the fractographic analysis shown in Fig. 8d. The minimum predicted fatigue strength is lower than the experimental fatigue endurance; hence crack initiation should be possible also at stress amplitudes lower than those explored experimentally in Fig. 7a. This could explain the behaviour displayed by the shot peened variant, viz. an increment in fatigue strength with respect to the as-built condition accompanied by a steadily declining SN curve. Accordingly, cracks are invariably destined to nucleate below the treated layer, even though at a higher stress level compared to the as-built condition; after initiation, cracks are forced to propagate up to the sample surface passing through a

strong compressive residual stress field, which slows down their growth rate, resulting in a further extension of the component lifetime.

Similar considerations hold for the HIPed variant. Work hardening and compressive residual stresses prevent crack initiation from the largest pores located immediately beneath the outer surface. Crack initiation occurs then, at least in the very high cycle fatigue regime, at pores located immediately below the hardened surface layer, as confirmed by the SEM micrograph shown in Fig. 8e. Their size is particularly small, on the order of 20÷30 μm, and their effect on the fatigue strength is not well captured by Eq. (7). In fact, the minimum predicted fatigue strength is significantly lower than the experimental one. This discrepancy cannot be explained in the same way as the peened variant, since the SN curve of the HIPed condition displays an asymptotic behavior with increasing lifetime. Apparently, when the defect size becomes very small, its effect on the fatigue strength is less detrimental than that predicted by Eq. (7). The reduced defect sensitivity of the HIPed variant can be a result of the thermo-mechanical treatment, which confers higher ductility to the material, as also attested by the results of the monotonic tensile tests. Future work is needed to address this important topic.

In conclusion, the model devised in this section can be used to interpret the fatigue behaviour of some of the material variants investigated in the present work in comparison with fatigue results published in the open technical literature. The assessment is only qualitative, as the input data of the model (viz. hardness, residual stresses, maximum defect size) are often not available, yet provides useful points of discussion. Specifically, Figure 12a and b compare the fatigue data for fully reversed (R=-1) and nearly pulsating (R≈0) fatigue loading, respectively. Fig. 12a reports the fatigue data published by Kasperovich and Hausmann [22], Leuders et al. [25], and Günther et al. [28]. In all these papers, the samples were machined from cylindrical bars obtained via SLM, sometimes subjected to HIP. Machining is likely to remove the largest defects, usually located in the surface layers, and to introduce beneficial surface compression residual stresses. This results in higher fatigue strength with respect to the present as-built and HIPed condition. Moreover, the exceptionally high fatigue resistance reported in [25] for the HIPed specimens can be imputed to the very high static strength of the material (and consequently of the microhardness) and to the samples' hourglass geometry that minimizes the presence of critical defects in the gauge section. On the contrary, if the surface is not machined after SLM, as in [22], the HIPed condition displays fatigue strength lower than that found in

the present paper, presumably due to lower microhardness (320 vs. 340 HV) and higher surface roughness (not explicitly indicated in [22], but reasonably higher than in the present paper as a result of the coarser powder size). Looking at Fig. 12b, it can be noted that the present as-built condition displays superior fatigue strength than that found by Edwards and Ramulu [26], since in the latter case the samples display after SLM a much larger surface roughness (Ra 33 μm), lower yield stress (and thus lower microhardness), tensile surface residual stresses. Same considerations hold for the comparison with the results reported by Wycisk et al. [21], where the as-built condition is affected by higher surface roughness (Ra 12 □m) and larger defects (as it can be inferred from the SEM fractrographic analyses). Conversely, the present as-built condition performs worse than the machined variants of Liu et al. [20] and [21] due to machining induced surface compressive residual stresses, removal of surface defects and hourglass sample configuration.

From the comparison with the literature data, we can finally infer that the observation made in Section 3.5 about the marginal role played by surface roughness in dictating the fatigue behaviour is reasonable only as long as the surface roughness is kept below the relatively low values reported in the present paper. Conversely, when the surface roughness becomes significant (Ra > 10 μm), its influence on fatigue becomes important, presumably due to synergistic effects with the defectiveness of the surface layers. In view of this observation, the Murakami model expressed by Eq. (7) might be further refined incorporating the surface roughness into the definition of the $\sqrt{area}$ parameter [29].

5. Conclusions

The low and high cycle fatigue behaviour of Ti-6Al-4V ELI additively manufactured via Selective Laser Melting was investigated on samples that received a low temperature stress-relief treatment. The effect of selected post processing treatments on the high cycle fatigue response was explored. Material characterization was complemented with residual stress and microhardness measurements, computed tomography scans, metallographic and fractographic inspections. The following conclusions can be drawn:

1) The as-built condition shows high tensile strength combined with sufficient ductility. The material undergoes significant cyclic strain softening. Even at the highest explored strain amplitudes, the strain is predominantly elastic. The LCF behavior is well represented by Basquin-Coffin-Manson equation, which can be extended up to the high cycle fatigue regime.

2) The high cycle fatigue regime is mainly dictated by mean stress and defectiveness. The former effect is well captured by the SWT model, thus suggesting that the fatigue response is strongly influenced by the maximum stress. The latter effect is well represented by the Murakami $\sqrt{area}$ model.
3) In the as-built condition, the highest porosity (about 0.35%) and largest defects are located in the outermost 0.4 mm thick layer and are preferential crack initiation site. HIP is effective in reducing the porosity throughout the sample (below 0.05%), while shot peening changes the shape rather than the volume of the pores located in the outermost layer.
4) The high-cycle fatigue resistance of the as-built condition is about 20% of the material tensile strength, thus significantly inferior to that of conventionally processed Ti-64.
5) Electropolishing is little effective in enhancing the fatigue strength, as it exposes critical defects on the surface. Therefore, the surface finish seems to exert a negligible influence on the fatigue resistance of this material, at least as long as the surface roughness of the as-built condition is kept below the values reported in the present paper.
6) Work hardening and compressive residual stresses due to shot peening prevent crack initiation in the surface layer but not the initiation of sub-surface cracks in the very high cycle fatigue regime. This results in a steadily declining SN curve.
7) Very small defects remaining after HIP seem to be less detrimental to the fatigue response than predicted by the Murakami $\sqrt{area}$ model. Further investigations are required to understand the material modifications produced by this thermo-mechanical treatment.

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Tables

Table 1. CT scanning parameters

Parameter	**Value**
Voltage	170 kV
Current	41 µA
Power	7 W
Exposure time	2000 ms
Nr. of projections	2500

Table 2. Monotonic tensile properties of the Ti-6Al-4V ELI alloy.

Condition	**E (GPa)**	**$\sigma_{Y0.2}$ (MPa)**	**UTS (MPa)**	**T.E. (%)**
As-built	113	1015	1090	10
Annealed	110	880	950	11
HIPed	110	850	960	14

E: Young's modulus, $\sigma_{Y0.2}$: 0.2% yield stress; UTS: ultimate tensile strength; T.E.: total elongation.

Table 3. Surface roughness properties.

Condition	**Ra (µm)**	**Rz (µm)**	**R_{max} (µm)**
As-built	6.83	38.40	43.85
Electropolished	0.54	2.66	4.41
HIPed	5.07	28.10	35.50
Shot peened	3.36	16.48	20.05

Table 4. Coffin-Manson and Basquin parameters (Eq. (1)) of the LCF curve.

E **(MPa)**	σ'_f **(MPa)**	b	ε'_f	c
113000	3120	-0.186	15.35	-1.47

Table 5. Ramberg-Osgood parameters (Eq. (3)) of the stress-strain curve.

Curve	E **(MPa)**	H' **(MPa)**	n'
Cyclic	113000	1875	0.105
Monotonic	113000	1254	0.031

Table 6. Principal results of the fatigue tests.

Condition	**R**	**Equation**	c_1 **(MPa)**	c_2	m	S **(MPa)**
As-built	-1	(1b)	221	123927	0.646	24.2
	-3	(1b)	308	22691	0.440	20.2
	0.1	(1b)	141	4.28435×10^6	1.035	10.6
Annealed	-1	(1b)	191	3087	0.384	11.9
Electropolished	-1	(1b)	242	8706	0.376	17.0
HIPed	-1	(1b)	369	2.30018×10^6	0.897	20.9
Shot peened	-1	(1a)	1315	13.1	-	19.4

Figures captions

Fig. 1. Sample geometry used for push-pull fatigue tests (standard ASTM E606). Dimensions are in mm.

Fig. 2. Light optical micrographs of the microstructure of the Ti6Al4V ELI SLM sample. (a) As-built condition. Annealed for 2h at (b) 900°C, (c) 920°C and (d) 950°C. (e) after the HIP treatment.

Fig. 3. Scheme of geometrical characteristics of pores detected by CT scans.

Fig. 4. (a) Microhardness profiles, obtained by averaging the results of three tests. (b) Residual stress profiles measured by XRD technique. The stabilized residual stresses are measured on a shot peened sample fatigued at stress amplitude of 350 MPa for 32×10^6 cycles.

Fig. 5. Results of the defectiveness analysis. (a) Annular areas in which the cross-section is divided. The image is obtained by collating several optical micrographs (b) Comparison of the porosity estimated from metallographic inspection and CT scans. (c) Average value of the non-dimensional defect shape factor ϕ defined by Eq. (6) based on CT scans.

Fig. 6. Principal results of the LCF tests. (a) Evolution of the stress amplitude vs. number of strain cycles. (b) Stabilized hysteresis loops at half fatigue life. (c) Experimental data vs. Basquin and Coffin-Manson equations. (d) Comparison between monotonic and cyclic stress strain curve.

Fig. 7. Axial fatigue SN curves. (a) Reverse (R = -1) fatigue tests carried out on all the investigated material variants. (b) Fatigue tests carried out on the as-built condition at three different stress ratios. Run-out tests are marked by arrows. (c) Haigh diagram showing the effect of mean stress on the as-built condition.

Fig. 8. SEM micrographs of the fracture surfaces around the fatigue crack initiation site. The defect responsible for crack nucleation is indicated by a red arrow and circumscribed by a dashed line. (a) as-built (σ_a= 250 MPa, N_f =

1.31×10^5), (b) annealed (σ_a= 200 MPa, $N_f = 5.93\times10^6$), (c) electropolished (σ_a= 275 MPa, $N_f = 3.97\times10^6$), (d) shot peened (σ_a= 350 MPa, $N_f = 2.91\times10^7$), (e) HIPed (σ_a= 375 MPa, $N_f = 7.1\times10^6$), (f) HIPed (σ_a= 425 MPa, $N_f = 9.95\times10^4$) sample. All the samples are tested under reversed axial tests.

Fig. 9. (a) Cumulative probability distributions of defect dimension $\sqrt{area}$. (b) Distribution of the maximum expected defect size as a function of the depth below the surface.

Fig. 10. Calibration of the Murakami $\sqrt{area}$ model (Eq. (7)) using the high cycle fatigue strength data of the as-built and annealed conditions.

Fig. 11. Predicted fatigue strength vs. depth below the surface for (b) as-built and electropolished, (c) shot peened and HIPed conditions.

Fig. 12. Comparison of the present fatigue results with data found in the open technical literature. (a) fully-reversed (R=-1) and (b) nearly pulsating ($R\approx0$) fatigue loading. Run-out tests are marked by arrows. The dashed lines indicate the [-S,+S] scatter band around the 50% failure probability fatigue curve expressed by Eq. (4).