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Influence of layer thickness and post-process treatments on the fatigue properties of CoCr scaffolds produced by laser powder bed fusion

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Abstract

Over the last years, additive manufacturing (AM) techniques such as laser powder bed fusion (L-PBF) have been frequently adopted for efficiently producing biomedical implants. L-PBF offers the advantage of low material waste and high accuracy enabling the production of complex and highly personalized geometries. However, when manufacturing time is considered, the L-PBF production rate is relatively low compared to conventional production techniques. The aim of this paper is to present the impact of layer thickness on static and fatigue properties of CoCr scaffolds produced by means of L-PBF. An increased layer thickness (from 30 μm to 60 μm) leads to an improvement in terms of production rate of 40 to 50% without affecting the final geometry of the structure. A fatigue test campaign was conducted on both 30 μm and 60 μm layer thickness samples in "as-built" condition. The analysis of the test results with a local stress method highlighted no significant differences in terms of fatigue performances. In addition, the effect of post-process treatments, such as hot isostatic pressing (HIP) and chemical etching on static and fatigue properties were investigated. It is shown that HIP does not affect the fatigue properties of the scaffolds whilst chemical etching is capable of improving fatigue resistance when the local stress approach is considered.

Keywords: laser powder bed fusion, CoCr, fatigue, layer thickness, (increased) productivity

1. Introduction

With their excellent biocompatibility and high resistance to corrosion and wear, CoCr alloys are extensively used for biomaterial applications, such as orthopedic implants and dental devices [1]. When considering such biomedical

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components, the possibility of designing and manufacturing porous structures is particularly relevant. Indeed, the incorporation of porous scaffolds as a substitute to human bone is known to favor bone ingrowth and implant anchorage [2] [3]. Moreover, in order to avoid stress shielding, subsequent bone resorption and implant loosening [3], the relative density of these porous structures can be specifically designed so that their stiffness matches that of the bone. Hence, long-term osseointegration of the implant is enhanced [2].

Additive manufacturing (AM) processes, such as laser powder bed fusion (L-PBF) also frequently referred to as selective laser melting (SLM), are particularly suited for the production of complex geometries with tailored mechanical properties. Customized, patient-specific implants can be designed "on-demand", which provides a better fit in the implant site, shorter surgery times, a faster recovery and reduced delivery costs [4]. SLM of CoCr dental [5] [6] [7] [8] [9] [10] [11], orthopedic [12] and cardiovascular [13] devices as well as of CoCr scaffolds [3] [14] [15] has already been widely investigated.

Biomedical implants typically experience cyclic loading. Understanding their fatigue behavior is therefore very important in order to validate their long-term use in the human body. Several papers investigated the fatigue of scaffolds manufactured by SLM [16] [17], including that of CoCr scaffolds [14] [18] [19].

However, a major drawback of powder based AM techniques is their relatively low productivity, which directly affects their competitive advantage in the medical industry. This issue can be addressed by increasing the layer thickness, as already demonstrated on various materials, including Ti-6Al-4V [20], Inconel 718 [21], stainless steels [22] [23] and aluminum alloys [24]. By finely adjusting the process parameters, an increase in layer thickness can be implemented without impacting the material's relative density. However, the increase in productivity typically comes along with a reduced accuracy and a higher surface roughness [20] [23]. This can be very detrimental to fatigue properties, especially when considering geometries with a high surface-to-volume ratio, as is the case for scaffolds. To this day, the effect of increasing the layer thickness on the mechanical properties of L-PBF scaffolds has not been published in scientific literature.

In this work, the layer thickness of CoCr scaffolds manufactured by L-PBF is varied from $30\mu\text{m}$ to $60\mu\text{m}$, resulting in a 40 to 50% reduction in processing time. The impact of this increase in layer thickness on relative density, microstructure, surface finish, quasi-static properties and fatigue properties is evaluated in detail. This work also investigates the influence of two post-process treatments, namely Hot Isostatic Pressing and chemical etching, on the compressive and fatigue behavior of the scaffold.

2. Materials and methods

CoCr scaffolds were produced by 3D Systems, on a *ProX DMP 320* machine, using an ASTM F75 grade gas atomized powder as starting material (LaserForm CoCrF75 (A), 3D Systems). Cylindrical specimens were built vertically, i.e. with their longitudinal axis perpendicular to the build plate. The samples were designed using a diamond unit cell which has proven to have symmetric mechanical behavior when axial compressive and tensile quasi-static loads are applied to the structure [25].

The geometry and dimensions of the scaffold and unit cell are reported in Figure 1.

Two distinct layer thicknesses were used, namely $30\mu\text{m}$ and $60\mu\text{m}$, and the process parameters were adjusted accordingly. In both cases, the processing conditions were optimized so that dense struts with a very limited amount of residual porosity were produced. In total, 120 samples were built with a layer thickness of $30\mu\text{m}$ and 30 with a layer thickness of $60\mu\text{m}$. Hot Isostatic Pressing (HIP), i.e. heating the parts at 1200°C at a pressure of 1000bar for 4h , was performed on 60 $30\mu\text{m}$ layer thickness specimens. In order to investigate the impact of surface roughness on fatigue properties, chemical etching was performed on 30 HIP'ed and on 30 as-built (AB) $30\mu\text{m}$ layer thickness samples. The scaffolds were dipped in an aqueous solution containing 27 vol% HCl and 8 vol% H_2O_2 , i.e. an etchant specifically developed for CoCr scaffolds [14]. Overall, 5 processing and post-processing conditions were investigated: $30\mu\text{m}$ - AB, $30\mu\text{m}$ - AB - etch, $30\mu\text{m}$ - HIP, $30\mu\text{m}$ - HIP - etch and $60\mu\text{m}$ - AB.

For each of the above conditions, Archimedes' density measurements were performed on all the samples. For each sample dry weighting and weighting in pure ethanol was performed. Based on these two measurements and on the theoretical density of $8.35\text{g}/\text{cm}^3$ for CoCr, the strut density was calculated [26] [27]. An O'Haus Pioneer balance with an accuracy of 0.1mg equipped with an calibrated O'Haus Archimedes kit was used for gravimetric measurements.

The effect of chemical etching and layer thickness on surface finish was qualitatively assessed by performing SEM analysis of the struts. From both $30\mu\text{m}$ - AB and $60\mu\text{m}$ - AB batches, one sample was analyzed with a *Nikon XT H 225*

Figure 1: Dimensions of the cylindrical specimens and diamond unit cell.

ST computer tomography system to quantify the difference in geometry between the designed and printed scaffolds. The scanning was performed using a W target with a 1.5mm Cu filter, and the machine was set to a voltage of 170kV and a current of 150 μ A. The voxel size was 13.5 μ m. Data analysis, stl reconstruction, alignment and comparison were performed with VGSTUDIO and GOM inspect software.

The effect of layer thickness and HIP on the microstructure was investigated by optical microscopy (OM). For metallographic analysis, the scaffolds were cut along the build direction, ground and polished in accordance to standard metallographic procedures, with colloidal silica (OP-S) as a final polishing step. Electro-chemical etching of the surface was done by dipping it for 30s in a 10% HCl Solution and at a voltage of 3V.

Mechanical properties were evaluated by performing quasi-static compression tests and fatigue tests. For quasi-static tests, three specimens were tested in each condition according to the ISO 13314 standard [28] on a SHIMADZU AG-X Plus machine, at a constant displacement rate of 0.9mm/min. The displacement was measured by three linear variable differential transformers (LVDT). For fatigue tests, an INSTRON Electropuls E10000 machine was used. The tests were performed at room-temperature, with a constant amplitude sinusoidal loading in compression-compression ($R = 10$). 5 stress levels were investigated, with σ_{min} equal to -40, -35, -30, -25 and -20 MPa. For each σ_{min} value, at least two specimens were tested, at a frequency of 30Hz. Fatigue tests were stopped when one of the two following conditions occurred: permanent plastic deformation of 1mm or failure of the specimen. If neither of these criteria was met, the tests were stopped at 2×10^6 cycles (run-out).

From the fatigue test results, two types of $S-N$ curves were obtained. In the first, the global stress, i.e. the applied force divided by the cross-sectional area of the scaffold was plotted as a function of the number of cycles to failure. For a better understanding of the material's behavior at the scale of a strut, the local stress method developed by Van Hooreweder et al. [16] was used as well. During compression of the scaffold, the total stress in a strut is considered as the superposition of an axial compressive stress and a bending stress, as indicated in Figure 2. For fatigue, the tensile

Figure 2: Local stress calculation approach in a strut.

stress component is more critical than the compressive stress component [16]. Based on the following equation, the maximum normal tensile stress in the struts was calculated:

$$S_1 = F \frac{16L \cos \theta}{\pi d^3} - F \frac{4 \sin \theta}{\pi d^2} \quad (1)$$

with F being the strut force, d the strut diameter, $\theta = 35.26^\circ$ the orientation of the strut force and L the strut length.

3. Results

3.1. Relative density

The relative density of the struts, ρ_{strut} , and of the whole structure, $\rho_{structure}$, measured by the Archimedes' method, are reported in Table 1.

Table 1: Relative density of the struts and of the structure for a layer thickness of $30\mu\text{m}$ in the as-built, etched, HIP, HIP and etched conditions and for a layer thickness of $60\mu\text{m}$ in the as-built condition

Condition	$\rho_{strut}[\%]$	$\rho_{structure}[\%]$
$30\mu\text{m}$ - AB	99.70 ± 0.03	22.90 ± 0.27
$30\mu\text{m}$ - AB - etch	99.65 ± 0.05	19.08 ± 0.76
$30\mu\text{m}$ - HIP	99.95 ± 0.03	22.95 ± 0.19
$30\mu\text{m}$ - HIP - etch	100.06 ± 0.04	17.39 ± 0.45
$60\mu\text{m}$ - AB	99.66 ± 0.07	24.76 ± 0.25

Even in their as-built condition, and independently of their layer thickness, the struts have a low volume fraction of residual porosity, with ρ_{strut} being systematically larger than 99.50%. The HIP post-treatment proves to be efficient in further decreasing the amount of residual porosity. Their volume fraction is reduced to less than 0.1% after HIP.

The relative density of the scaffold $\rho_{structure}$ is directly related to the processing conditions, in this case the layer thickness, and to the surface post-treatments. When the layer thickness is increased, the relative density of the scaffold $\rho_{structure}$ is also increased by about 2%. On the other hand, etching, by removing material from the struts surface, results in a decrease of the scaffold's relative density.

Figure 3: Optical micrographs of a transverse cross-section, for a layer thickness of $30\mu\text{m}$ (a, b), for a layer thickness of $60\mu\text{m}$ (c, d) and for a layer thickness of $30\mu\text{m}$ with HIP (e, f). The arrow indicates the build direction.

3.2. Microstructure analysis

As illustrated in the optical micrographs in Figure 3, the as-built material exhibits overlapping arc-shaped melt pool boundaries resulting from fast solidification of the melt pool during the production process. It can be noticed that the inter-boundary spacing is directly related to the layer thickness. Fine cellular dendrites ($0.5 - 1\mu\text{m}$ thick) grow epitaxially within the melt pools, until they impinge cells growing in a more favorable direction, i.e. closer to that of the temperature gradient direction. This microstructure, which does not seem to be influenced by the layer thickness, has already been extensively described by various authors [6] [11] [19] [29] [30].

HIP induces a complete recrystallization of the grains (Figure 3e and 3f), resulting in a much more isotropic microstructure. The melt pool boundaries are no longer visible after HIP. Similar results were previously observed in Co-Cr parts built either by SLM [31] or by EBM [30].

3.3. Morphological characterization

Figure 4: SEM micrograph of the surface of (a) as-built and (b) etched struts built with a layer thickness of $30\mu\text{m}$ and (c) as-built struts built with a layer thickness of $60\mu\text{m}$.

SEM analysis of the struts allows their surface finish to be qualitatively investigated. The comparison of the SEM micrographs in Figure 4a and Figure 4b reveals an improvement in surface finish with chemical etching. The small powder particles attached to the surface are preferentially removed by chemical etching. However, this comes along with a micro-pitting of the surface, which can be seen in Figure 4b.

In Figure 4c the scaffold built with a higher layer thickness exhibits larger melt pools, which visibly impacts the morphology of the struts.

For a more quantitative analysis of the impact of layer thickness on the final geometry CT scans were performed on both $30\mu\text{m}$ - AB and $60\mu\text{m}$ - AB scaffolds. In Figure 5a and 5b are presented the surface deviation between the reconstructed scan images and the CAD input file of a few unit cells from both $30\mu\text{m}$ - AB and $60\mu\text{m}$ - AB samples. From these images, it is clearly visible that the greatest deviation occurred at the location of the nodes (intersection

Figure 5: (a) Map of surface deviation of 30 μm - AB scaffold from CAD (blue); (b) Map of surface deviation of 60 μm - AB scaffold from CAD (blue); (c) distribution of reconstructed surface of 30 μm - AB and 60 μm - AB sample in function of their distance from the CAD model.

of four struts) and along the down-facing strut surfaces leading to a higher roughness when 60 μm - AB samples are compared with 30 μm - AB samples. The deviation between the actual and the designed structure can be attributed to dross formation along the down-facing surfaces.

The curves in Figure 5c indicate the CT reconstructed surfaces in function of their distance from the CAD file. For the 60 μm - AB sample a slightly bigger surface has a positive deviation from the CAD geometry compared to the 30 μm - AB sample.

Despite these differences, Figure 5a and b show that both 30 μm - AB and 60 μm - AB scaffolds properly replicate the designed geometry.

3.4. Quasi-static compression

Figure 6: Averaged quasi-static compression stress-strain curves for each of the different processing and post-processing conditions

Quasi-static compression testing was performed for all five processing and post-processing conditions. The results are reported in Figure 6 and in Table 2. As-built specimens exhibit a higher strength, but a lower ductility than HIP'ed specimens. Etched specimens have lower compressive properties than their as-built counterparts.

Table 2: Overview of the compressive properties obtained in various processing and post-processing conditions

Condition	Quasi elastic gradient [GPa]	Offset stress (0.2%) σ_y [MPa]	First maximum stress σ_{max} [MPa]	Elongation at σ_{max} [%]	Plateau [MPa]
30 μ m - AB	2.06 $\pm$ 0.33	36.07 $\pm$ 6.16	60.81 $\pm$ 3.07	15.13 $\pm$ 1.02	55.14 $\pm$ 2.03
30 μ m - AB - etch	1.56 $\pm$ 0.08	31.34 $\pm$ 2.31	45.32 $\pm$ 2.70	11.60 $\pm$ 1.22	42.23 $\pm$ 2.39
30 μ m - HIP	2.06 $\pm$ 0.25	33.15 $\pm$ 1.65	59.02 $\pm$ 1.67	23.47 $\pm$ 0.42	58.03 $\pm$ 1.96
30 μ m - HIP - etch	1.37 $\pm$ 0.15	22.42 $\pm$ 0.26	35.62 $\pm$ 1.42	16.00 $\pm$ 1.14	34.22 $\pm$ 1.19
60 μ m - AB	2.34 $\pm$ 0.13	41.62 $\pm$ 1.54	62.82 $\pm$ 0.88	12.40 $\pm$ 0.46	57.09 $\pm$ 0.32

3.5. Fatigue testing

In Figure 6 the results of fatigue tests on all the different conditions are presented in terms of global stresses in function of the number of cycles to failure. By using the global approach, the differences in fatigue properties shown in Figure 7 are mainly caused by differences in relative density between different batches. As a result, the fatigue resistance of the samples with high relative density is higher compared to the samples with low relative density and the individual effect of post treatments and layer thickness cannot be appreciated. However, when the local stress approach is considered, the effect of chemical etching on fatigue properties is clearly visible (Figure 8).

Figure 7: S-N curves with failure data of as-built (AB), HIP and/or etched specimens built with a layer thickness of 30 μ m or 60 μ m, plotted on a semi-log scale using the global stress method. Figure (a) to (d) provide a straightforward comparison between different processing and post-processing conditions.

Figure 8: S-N curves with failure data of as-built (AB), HIP and/or etched specimens built with a layer thickness of 30μm or 60μm, plotted on a semi-log scale using the local stress method.

4. Discussion

Overall, it should be highlighted that optimized process parameters were used for both layer thicknesses in this study. As a result, the amount of residual porosity generated during the process (measured by means of Archimedes' principle) was systematically below 0.4%. In comparison, previous work reported up to 2% residual porosity in SLM CoCr scaffolds [14].

4.1. Effect of chemical etching

As can be seen in Figure 4, the surface finish of the struts is influenced to a large extent by processing and post-processing conditions. Shape of melt pool is directly related to laser power, which is typically higher for larger layer thickness, resulting in a long range waviness of the struts. The adhesion of partially melted powder particles to the surface during processing generates a significant mid-range roughness. On an even shorter range, micro-pitting induced by chemical etching creates small dimples on the surface of the struts.

Chemical etching has a twofold effect on surface roughness. On the one hand, it partially reduces the long-range waviness caused by the mismatch between successive melt pools, and also tends to substantially decrease the mid-range roughness, through the near-complete removal of powder particles stuck to the surface. These particles do not contribute to the scaffolds mechanical properties. Therefore, the fatigue resistance, evaluated with the local stress method, is substantially improved by chemical etching (Figure 8) due to the improvement of the mechanical efficiency of the structure. Furthermore, the removal of partially molten particles may help to reduce the risk of metallosis, i.e. adverse biological reactions due to the exposure to small particles resulting from wear of the implant [32].

On the other hand, chemical etching increases the short-range roughness by inducing micro-pitting of the surface. When considering biomedical applications, this can prove to be beneficial. According to Mustafa et al., surface roughness of the implant material favors cellular attachment and proliferation [33]. In other words, this short-range, moderate surface roughness could enhance long-term osseointegration, without being detrimental to the mechanical properties.

On a global scale, chemical etching results in a decrease of the struts diameter, and therefore of the scaffold's relative density. As reported in Table 1, after chemical etching, $\rho_{structure}$ is about 4% lower than in the as-built condition. Consequently, the etched structure experiences a lower quasi-static strength (Figure 6) and a lower fatigue resistance (Figure 7c) than its as-built counterpart. In industrial production the scaffold dimensions should be designed to compensate for the reduction of strut diameter due to post processing.

4.2. Effect of HIP

As highlighted in Figure 3e and 3f, HIP has a substantial influence on the microstructure of CoCr scaffolds. Heating the material for 4h at a temperature of 1200°C results in complete recrystallization: the microstructure evolves

from very fine, epitaxially growing, cellular dendrites to coarser equiaxed grains. Furthermore the amount of internal porosity can be reduced during the HIP treatment.

HIP has an important impact on the quasi-static properties of CoCr scaffolds, as shown in Figure 6 and Table 2. HIP'ed specimens exhibit a lower strength and a higher ductility. This can be directly related to the coarse microstructure generated by full recrystallization during the treatment.

However, HIP only has a very moderate impact on the fatigue properties. A slight increase in the slope of the S-N curves of HIPed specimens with respect to as-built scaffolds can be observed in Figure 7b. This could be a consequence of the microstructural changes induced by the treatment that lead to an improvement in the ductility of the samples (Figure 6 and Table 2). This tendency was reported by Haan et al. for bulk CoCr specimens, although they also observed an important increase in fatigue resistance in HIP'ed samples [31].

The same trend of an increase in the slope of the S-N curve with HIP can be observed in the case of etched scaffolds (Figure 7c.). Here, the low fatigue properties obtained for the HIP'ed samples compared to the as-built ones (Figure 7c) can be directly related to the low relative density $\rho_{structure}$ of the HIP'ed scaffolds (Table 1). This scatter in relative density between "HIP and etched" and "AB and etched" samples is due to the variability in the amount of material removed from the scaffold during etching. The coarser microstructure generated after HIP process, might induce a change in the material corrosion behavior, leading to a lower $\rho_{structure}$ of the 30 μm - HIP - etch samples with respect to 30 μm - AB - etch specimens.

Overall, the limited impact of HIP on fatigue in the current study can be related to the very high relative density and lack of residual stresses of the as-built material. As a result, HIP only induces a very slight decrease in the amount of residual porosity, which is hardly significant for fatigue properties.

4.3. Effect of an increase in layer thickness

Due to a difference in thermal behavior, scan parameters were optimized for both 30 μm and 60 μm layer thickness to ensure an optimal balance between printed part density, printed part accuracy and productivity. Furthermore productivity dead time, such as minimal cooling time per layer and coating time, needs to be taken into account. By considering all the factors, doubling the layer thickness yields to an improvement in the productivity rate from 40 to 50%.

When the layer thickness is increased, deeper melt pools are generated, as illustrated in Figure 3. Despite this change in melt pool dimensions, the microstructure remains cellular and fine, with a sub-micron cell-spacing, so that the tensile properties are not directly impacted. It can indeed be seen from Figure 6 and Table 2 that strength and ductility are barely affected by a higher layer thickness.

Increasing the layer thickness directly impacts the geometry of the struts, which present a more important staircase effect and a higher amount of dross on the down-facing surface of the struts. As a result, 60 μm layer thickness scaffolds exhibit a lower accuracy and a higher surface roughness (Figure 5a, 5b and 4) than 30 μm - AB samples.

Besides, the struts tend to be larger, increasing the overall relative density of the scaffold. Therefore, a slightly higher quasi-static strength and global fatigue resistance of the structure are obtained (Figure 7a). When the local stress approach is considered, both 60 μm - AB and 30 μm - AB samples exhibit comparable fatigue performances (Figure 8) despite their difference in surface finishing and dimensional accuracy.

5. Conclusion

This study has shown that increasing the layer thickness by a factor two, provided that print parameters are optimized accordingly, can result in a significant productivity improvement of about 40% to 50% for CoCr parts. Despite a reduced geometrical accuracy, quasi-static and fatigue properties are not significantly affected.

Furthermore, the benefits of performing surface post-treatment such as chemical etching for the quasi-static and the dynamic properties of CoCr L-PBF scaffolds were clearly demonstrated.

Chemical etching results in a decrease in the overall density of the scaffolds, so that the strength and fatigue life are directly impacted. However, on a local scale, improving the surface finish of the struts through chemical etching results in a substantial increase in fatigue resistance. The treatment removes partially molten particles attached to the struts that are not contributing to the structure mechanical properties, indirectly improving the scaffolds fatigue performances when local stresses are considered.

A thermal post-treatment such as HIP has a large influence on the microstructure of the material, and consequently on the quasi-static properties of the specimens. HIP induces a desirable increase in ductility, but this comes along with a decrease in strength. However, in terms of fatigue properties, in the case of high-quality as-built parts with very low residual porosity, the effect of HIP remains limited.

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Local Stress [MPa]

Processing

Etch

+ [40 – 50]% productivity