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Procedia Structural Integrity 2 (2016) 3168–3176

Structural Integrity Procedia

www.elsevier.com/locate/procedia

21st European Conference on Fracture, ECF21, 20-24 June 2016, Catania, Italy

Fatigue characterization of Titanium Ti-6Al-4V samples produced by Additive Manufacturing

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Abstract

Additive Manufacturing offers real opportunities in Thales, since it enables a flexible and cost-effective production of metallic components directly from a 3D digital data model. The manufacture of the parts, layer by layer, allows building more complex geometries by adding new functionalities or reducing the weight. The products of Thales can be more competitive and attractive.
Studying and analysing the mechanical properties of such samples is essential. In order to be able to choose the eligible parts, design these parts and ensure their robustness, it is necessary to understand the static and fatigue behaviour of materials built by Additive Manufacturing and, more particularly, the damaging process regarding the microstructure.
Following a first study on the static properties of a Titanium alloy used within Thales, a large set of fatigue tests, including both HCF and LCF, have been performed to evaluate the fatigue performances of Ti-6Al-4V samples built by a powder bed fusion process. The fatigue mechanisms and the obtained lifetimes were compared with more conventional processes (casting, wrought).
In this paper, the results of these fatigue tests on Titanium (Ti-6Al-4V) samples will be presented. Different parameters have been compared: building orientation, heat treatment (HIP) and post-machining. The fracture mechanisms have also been analysed by performing a correlation between the microstructure analysis (porosity, metallography) and fractographies. It is shown that fatigue performances depend on the selected parameters but these effects are different, depending on the loading domain (HCF, LCF).

Keywords: Additive Manufacturing ; Fatigue ; Titanium

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Peer review under responsibility of the Scientific Committee of ECF21.
10.1016/j.prostr.2016.06.395

1. Introduction

Additive Manufacturing is the name for a group of processes which appeared in the 80's and consists in building parts layer by layer. A 3D model is directly sent to the machine which builds it (American Standard for Testing and Material International (2012)). These technologies were previously used to manufacture prototypes in plastics, but now they can build metallic and ceramic parts. Today, the objective is to manufacture functional parts (Reeves (2009)).

Manufacturing parts layer by layer allows a high freedom in design and this technology becomes really interesting to reduce weight and to add functionalities in parts. The more complex the part is, the more competitive this technology will be because the manufacturing time will be shorter and the price will be cheaper than for conventional processes. With all these advantages, many fields, including the aerospace industry, start to use this technology (Wohlers (2013)).

One of the technologies used to build metallic parts is the Selective Laser Melting (SLM) and it is described in detail by Kruth et al. (2007). It is a powder bed fusion process according to ASTM F2792 (American Standard for Testing and Material International (2012)) which means that a thermal energy selectively fuses regions of a powder bed. A powder bed is deposited on a plate and the selected zone is melted by a laser beam which brings the thermal energy. The next layer of powder is deposited on the previous one and the beam fuses this new layer. These steps are repeated until the final part is built.

In this study, Titanium Ti-6Al-4V has been processed. This Titanium alloy is the most used in the aerospace industry because of its high mechanical properties, low density and excellent corrosion protection properties (Boyer (1996)). In Additive Manufacturing, there are many examples of parts built with this alloy, and in order to industrialise the process and the use of this alloy, material health and mechanical properties must be mastered.

Many manufacturing parameters can influence the mechanical properties and the microstructure (Levy (2010)). The main parameters studied in the literature concern the powder (size, distribution, shape…) (Spierings et al. (2011)), the machine (layer thickness, beam power, scan speed, scanning strategy…) (Song et al. (2012)) and the post-treatments (cleaning, heat treatments, polishing…) (Vilaro et al. (2011)). Optimisation of all these parameters leads to parts with high mechanical properties and dense microstructure.

Microstructure and tensile properties on Ti-6Al-4V produced by SLM have already been deeply analysed. In SLM, there is an anisotropic microstructure with prior elongated grains oriented in a perpendicular direction to the layers. As-built parts have a martensitic structure (Vrancken et al. (2012)). The tensile properties are good compared to conventional processes (casting, wrought) except for the ductility which is a little lower. The anisotropy is negligible except for the ductility in favor of the Z axis or the X-Y axis depending on the source (Vilaro et al. (2011)) (Qiu et al. (2013)). After an HIP heat treatment, the microstructure is changed and is composed of thicker lamellar grains (Kasperovich and Hausmann (2015)) as found in more conventional processes (Nalla et al. (2002)). Performing an HIP heat treatment improves the ductility but lowers the mechanical resistance (Thöne et al. (2012)).

Some fatigue tests results are available in the literature, but the effects of the parameters on the fatigue life and the fracture mechanisms have not been analysed (Li et al. (2016)). The effects of surface roughness and post-heat treatments were studied in very few papers (Wycisk et al. (2013))(Thöne et al. (2012)). These types of tests and analysis are compulsory in the objective of the process industrialisation.

The aims of the study are to analyse the effects of several parameters on the fatigue properties of parts produced by SLM and to understand the fracture mechanisms. After presenting the materials and methods used in this study, the fatigue tests results, at high number of cycles (HCF) and low number of cycles (LCF), are exposed. In a last part, the fracture mechanisms are analysed by identifying the fracture initiation defects and their critical parameters. From the fatigue tests results, the effects of three parameters are observed: manufacturing direction, surface roughness and HIP heat treatment.

2. Materials and Methods

2.1. Material and process

The material used in this study is the Ti6Al4V titanium alloy. The chemical composition of this alloy is in accordance with the ASTM F2924 standard (American Standard for Testing and Material International (2014)). This standard concerns Ti6Al4V parts built by powder bed fusion technologies. However, there are no requirements on the fatigue performances of these parts, as in the standards for casting or wrought parts.

All the specimens were produced on an SLM 250HL machine with a layer thickness of 50µm. 2 types of cylindrical specimens were built for 2 types of tests: HCF (high cycle fatigue) and LCF (low cycle fatigue). The list of the specimens is reported in Table 1. Most of the specimens were produced in the Z axis, which means that loading is made in the normal axis to the layers. In the X-Y axis samples, loading is made in the direction parallel to the layers. All the parts were stress relieved at 650°C for 4 hours before cutting them from the base plate. An HIP heat treatment was performed on some of the specimens at 920°C and 1020 bars during 2 hours. After post-machining, the fatigue samples were polished by vibratory finishing in order to obtain a surface roughness Ra≈0.2µm in accordance with the ISO 1099 standard (International Organization for Standardization (2006)) for fatigue tests.

It has been considered that the specimens built in the Z axis, stress relieved and polished are the reference. All the specimens allow us to evaluate the effect of 3 parameters:

- Manufacturing direction
- Surface roughness
- HIP heat treatment.

Table 1. List of the specimens tested in this study

Type of test	Z axis 650°C/4h Machined/polished	X-Y axis 650°C/4h Machined/polished	Z axis 650°C/4h As-built	Z axis 650°C/4h + HIP Machined/polished
HCF test	12	12	12	12
LCF test	8	8	8	8

2.2. Testing methods

Optical microscopy observations were made on some specimens on the x-z plan using a Nikon Eclipse MA200. After testing, the specimens were cut in the two directions, polished and etched with a Kroll's etching solution in order to reveal the microstructure.

HCF tests were performed on an Amsler vibrophore, with constant stress amplitude and in accordance with the ISO 1099 standard (International Organization for Standardization (2006)). The tests were achieved with a stress ratio R=-1 (tension compression test) and a frequency around 90Hz. The test was stopped if no failure appeared after 10^7 cycles.

LCF tests were performed on an Instron 8800 hydraulic machine equipped with a contact extensometer Instron 2620, with constant strain amplitude and in accordance with the NF A03-403 standard (AFNOR (1990)). The specimens were loaded with a stress ratio R=-1 (tension compression test) and with a constant deformation speed of 0.056 s^{-1} corresponding to frequencies going from 1.1 to 2.8 Hz. The test was stopped if no failure appeared after 10^4 cycles.

After the tests, some fracture surfaces were observed by SEM on a Hitachi S-3600N microscope at 20 kV.

3. Results

3.1. Microstructure

Titanium Ti6Al4V is an α/β alloy with a microstructure strongly dependent on the manufacturing process and on the post-treatment. As mentioned above, SLM parts undergo high thermal cycles during manufacturing, going from the melting point to the temperature of the chamber in a very short time.

The microstructure observation on stress relieved and HIPed specimens are shown in Fig. 1 with different magnifications. Fig. 1a shows an anisotropic texture with elongated grains in the Z direction (normal to the layers). These grains are identified as prior β grains. They grow in this direction with the partial remelting of the previous layers when one layer is produced. After HIP, the same elongated grains can be seen, as the temperature of the treatment is under the temperature of β transus (1000°C).

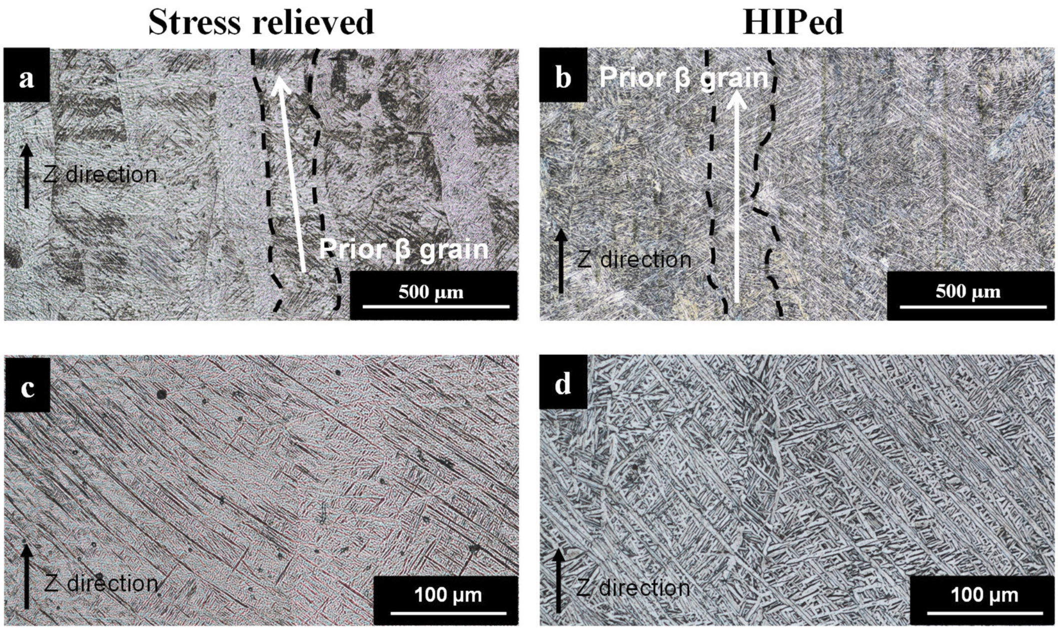

Fig. 1. Optical micrographs of SLM samples at different magnifications in the X–Z plane. (a,c): stress relieved (b,d): HIPed

A martensitic microstructure with acicular α’ grains which is characteristic for SLM parts is observed on stress relieved samples with a higher magnification (Fig. 1c). The anisotropic texture is no more visible. This type of microstructure explains the low ductility of these specimens (Vilaro et al. (2011)). It is fully changed after HIP and a Widmanstätten texture is observed with thick lamellar α grains well organized and very close to each other (Fig. 1d). They appear with the slow cooling in the HIP treatment, which allows the α grains to grow. An improvement in the ductility but a lower resistance results from this new microstructure (Thöne et al. (2012)).

3.2. Fatigue properties

Fatigue results of HCF tests (constant stress) are given on Fig. 2. The level of stress is given in relative values (relative to a stress value arbitrarily chosen) as this study is focused on the effect of the different parameters and not on the level of stress. The results are compared with data from the literature for casting and wrought processes on Fig. 2 (Welsch et al. (1993)).

From these results, 3 main observations can be made:

- The manufacturing direction has no major effect on the fatigue limit at 10^7 cycles. Due to the limited number of specimens, no clear difference can be observed at lower number of cycles.
- The surface roughness has a strong effect on the fatigue life of the specimens, with a fatigue life at 10^7 cycles which is doubled after machining and polishing post-treatments.
- The HIP heat treatment has a strong effect on the fatigue life at high number of cycles, but a limited effect at a lower number of cycles. The fatigue life at 10^7 cycles is improved by almost 90% after HIPing compared with stress relieved specimens.
- Stress relieved parts are at the level of casting and HIPed specimens are at the level of wrought processes at higher number of cycles.

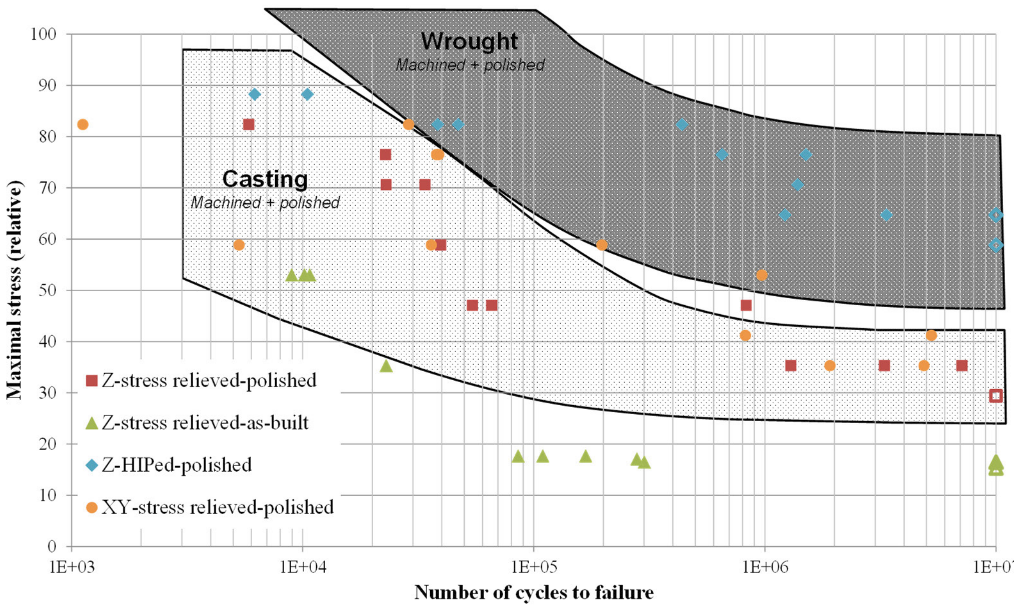

Fig. 2. Fatigue life of Ti6Al4V SLM specimens with different parameters. Unfilled points correspond to unbroken specimens after 10^7 cycles

LCF tests results are reported on Fig. 3. The total strain amplitude $\Delta\varepsilon_t/2$ is plotted against reversals to failure and is compared with the plastic strain $\Delta\varepsilon_p/2$ and elastic strain $\Delta\varepsilon_e/2$ with the relation given in equation (1). The four groups of specimens have been plotted in four different graphics. All the 8 specimens built for each group could not be tested because of the limited load cell used for this study, in particular for HIPed specimens. Plastic deformations below 0.01% are not taken into account as they are considered to be negligible.

$$\frac{\Delta\varepsilon_t}{2} = \frac{\Delta\varepsilon_p}{2} + \frac{\Delta\varepsilon_e}{2} \quad (1)$$

Most of the specimens experienced elastic strain with a very low plastic strain, especially the X-Y-stress relieved-polished (Fig. 3d) and Z-stress relieved-as-built (Fig. 3b) specimens. Z-stress relieved-polished samples (Fig. 3a) start to experience plastic strain at 0.9% total strain amplitude. Z-HIPed-polished specimens (Fig. 3c) experienced much more plastic strain and the two elastic and plastic curves are almost intersecting.

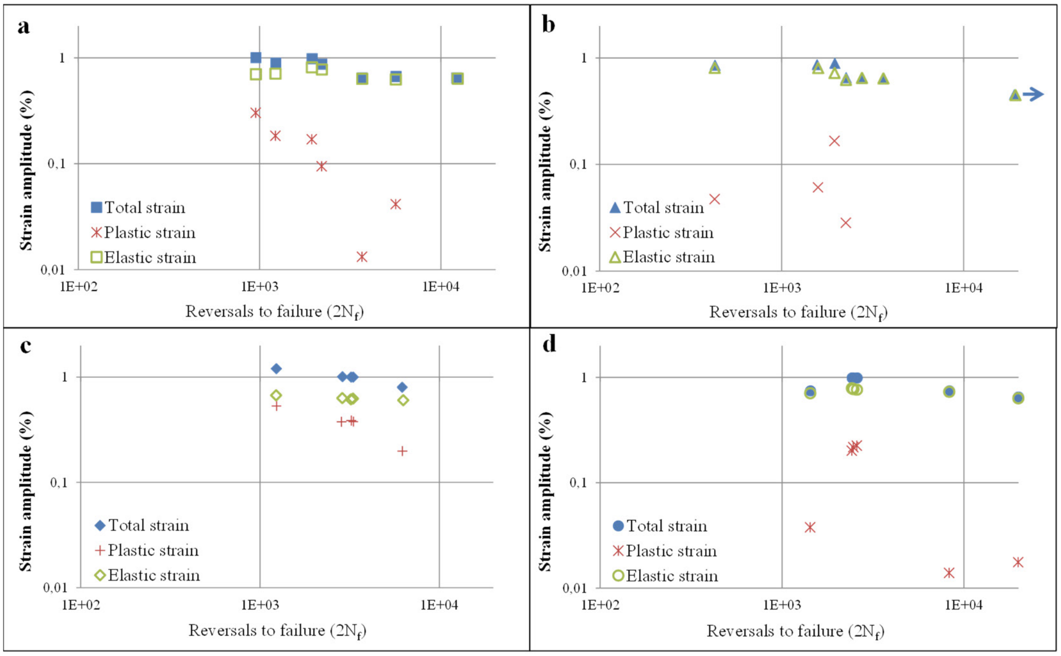

Fig. 3. Total, elastic and plastic strains against reversal to failure curves obtained after LCF tests for (a) Z-stress relieved-polished, (b) Z-stress relieved-as-built, (c) Z-HIPed-polished and (d) X-Y-stress relieved-polished specimens

4. Discussion

4.1. Fracture mechanisms

An examination of the fracture surfaces was completed on broken HCF and LCF tests samples in order to locate the fracture initiation zone and observe the fracture mechanisms depending on the parameters of the parts. On all the specimens, three types of fracture initiation defects have been observed and are illustrated on Fig. 4. Hereafter, they are ordered from the less critical to the more critical:

- **Porosities** are the first type of defects which initiates fracture on some specimens. They often come from trapped gas so they are spherical (Fig. 4a) (Gong et al. (2014)). The specimens which failed from porosities generally have a longer fatigue life.
- Some defects come from **unmelted zones** which mainly come from instability in the process. For example, the power of the laser beam can fluctuate and will induce a melting problem. Unmelted zones have different morphologies in the Z axis and X-Y axis fracture surfaces as in Fig. 4cd because they occur between two layers. This kind of defect seems to be more critical than porosities.
- **Surface defects** (emerging defects) seem to be the most critical for fatigue performances because they are favorable for a fissure initiation (Fig. 4b). These defects come from surface porosities, surface unmelted zones or simply from the high surface roughness of some parts.

On many samples, multiple fracture initiation sites have been observed on different types of defects, sometimes on different axial positions in the specimen. These samples show a fracture surface with different plates which correspond to the different initiation sites. They correspond to specimens which experienced a high stress amplitude in HCF tests and almost all the LCF tests samples.

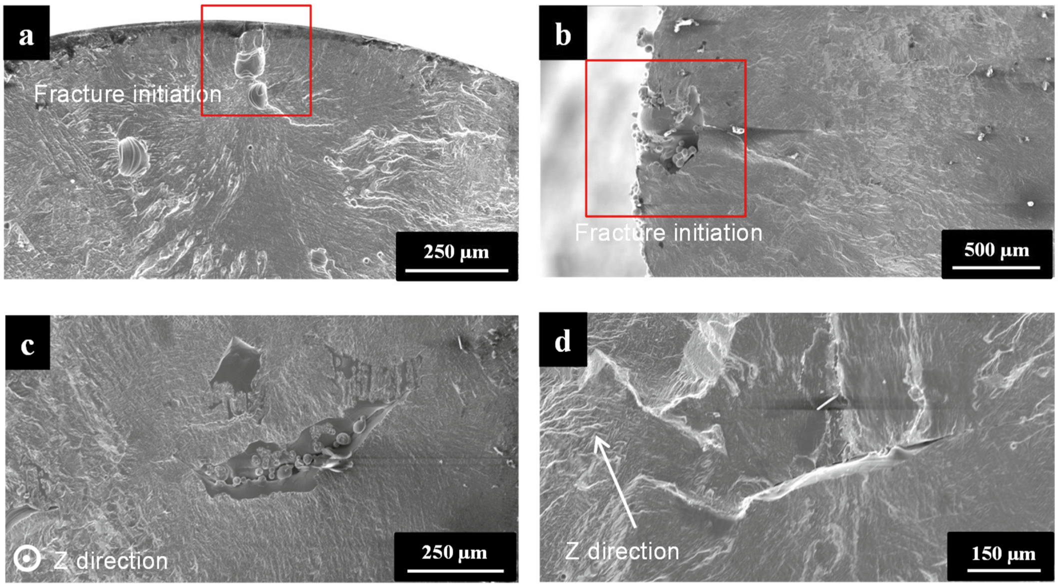

Fig. 4. Fractographs of the different types of defects (a) porosities, (b) surface defect (c) unmelted zone in the X-Y plan and (d) in the X-Z plan

Another observation made on the surface fracture is that the main rupture initiation defects were often placed close to the surface, at a distance less than 1 mm. Only a few of them were placed in the center of the parts. The defects are more critical when they are placed in this zone near the surface because the fracture is able to propagate easily and create emerging cracks.

Furthermore, no major effect of the amount of porosities on the fracture surfaces was observed. The position (defects on the surface are more critical), the size (big defects are more critical) and the concentration of the defects (a high concentration of defect on a specific zone is more critical) are the most important criterions favoring crack initiation to be considered.

Dispersion in fatigue life can be explained by these different types of fracture mechanisms depending on the specimen, its parameters and the loading.

4.2. Effects of the parameters

The effect of the surface roughness is clearly visible on the HCF curves and LCF curves. All of the as-built specimens failed from surface defects because of the high surface roughness of these samples (average roughness Ra≈ 15 µm) (Qiu et al. (2013)).

The low ductility of as-built specimens explains the negligible plastic strain in the LCF tests. It is typical for the martensitic microstructures of Ti6Al4V alloys in additive manufacturing.

The effect of the HIP post heat treatment is also clearly observed. After an HIP heat treatment, most of porosities and unmelted zones are closed or reduced. Due to this low amount of small defects, the samples have longer fatigue life than stress relieved specimens, especially at a high number of cycles. Examination of the fracture surfaces on these specimens confirmed that they failed from small porosities. No failures from unmelted zones were found in these samples.

As explained previously, HIP improves ductility and reduces the resistance compared to as-built and stress relieved SLM parts, due to the Widmanstätten pattern. This is confirmed by the higher plastic strain in the LCF tests compared to the stress relieved specimens. The lower resistance explains that the effect of this treatment is less visible at lower number of cycles.

No clear conclusion can be taken on the effect of manufacturing direction regarding Fig. 2. The number of specimens by level of stress is not sufficient to distinguish the two curves and the dispersion in the results for specimens with the same parameters is higher than the dispersion in the results between two different directions. However, the fatigue limit at 10^7 cycles for the two directions seems to be equivalent. The specimens near this limit failed from porosities. A difference can appear on specimens which failed from unmelted zones because of different morphologies depending on the direction (Fig. 4cd). In the Z axis samples, the stress concentration around the unmelted zones may be higher because of their orientations. In conclusion, specimens which contain no defaults, especially unmelted zones, should have isotropic fatigue life.

Concerning the LCF tests, the anisotropy in the ductility between the two directions explains the higher plastic strain of the Z axis samples. Indeed, the anisotropy in the ductility of SLM parts has often been observed in several articles (Qiu et al. (2013)) and is explained by the elongated prior β grains observed in the microstructures. There are more grain boundaries in the X-Z and Y-Z directions of loading than in the Z direction and ductility is lower.

5. Conclusion

In this study, the effect of three parameters on the fatigue properties and damage mechanisms of parts was analysed. The critical parameters of defects were also identified through fracture surfaces analysis.

The effects of surface roughness and HIP heat treatment were clearly observed in both HCF and LCF tests. The HIP reduces the amount and size of defects and thus, improves the fatigue life. Ductility was improved because of the different microstructure obtained after this treatment. On the contrary, the high roughness of as-built specimens involves a high amount of surface defects which were identified to be the most critical for fatigue performances. No major differences were found between the two manufacturing directions as the dispersion in one batch is higher than the dispersion between two different directions. It can be considered that there is no major anisotropy in the fatigue properties for parts which show no big defaults as unmelted zones. However, the anisotropy in ductility was confirmed by the LCF tests and was explained by the anisotropic texture of the microstructure.

Different types of defects were identified and classified from the less critical to the more critical for the fatigue life. These defects were observed on the fracture surfaces and a classification was made by comparing with the fatigue curves. The other parameters which are identified are the position, the size, and the concentration of these defects. Most of the fracture initiation defects were placed in a zone close to the surface, on the biggest one and on a concentration of defects when there was one.

To conclude, fatigue life of a part built by SLM is difficult to predict because of the different type of defects which can initiate a fracture. However, polishing and HIPing parts improve their properties. Methods to control the process are under development in order to predict the place and size of the defects and even to avoid their presence. With these methods, fatigue life could be easier to predict. However, fatigue properties of Titanium Ti6Al4V parts built by SLM are comparable to casting after stress relieving and comparable to wrought processes after an HIP heat treatment. A quantification of the size and position of the defects should be made in order to predict more precisely the fatigue life of SLM parts. The effect of the manufacturing direction should also be deeply studied.

Acknowledgements

The authors acknowledge Julien Moryousef for his help and support in the realization of some tests in the context of his project for his engineer degree course at Ecole Centrale de Lille.

References

AFNOR, 1990. NF A 03-403 Pratique des essais de fatigue oligocyclique.

American Standard for Testing and Material International, 2012. ASTM F2792: Standard Terminology for Additive Manufacturing Technologies.

American Standard for Testing and Material International, 2014. ASTM F2924 - Standard Specification for Additive Manufacturing Titanium-6 Aluminum-4 Vanadium with Powder Bed Fusion.

Boyer, R., 1996. An overview on the use of titanium in the aerospace industry. Materials Science and Engineering: A, 213(1), 103-114.

Gong, H. et al., 2014. Analysis of defect generation in Ti–6Al–4V parts made using powder bed fusion additive manufacturing processes. Additive Manufacturing, 1, 87-98.
International Organization for Standardization, 2006. ISO 1099 Metallic materials — Fatigue testing — Axial force-controlled method.
Kasperovich, G., Hausmann, J., 2015. Improvement of fatigue resistance and ductility of TiAl6V4 processed by selective laser melting. Journal of Materials Processing Technology, 220, 202-214.
Kruth, J.-P., Levy, G., Klocke, F., Childs, T., 2007. Consolidation phenomena in laser and powder-bed based layered manufacturing. CIRP Annals-Manufacturing Technology, 56(2), 730-759.
Levy, G. N., 2010. The role and future of the laser technology in the additive manufacturing environment. Physics Procedia, 5, 65-80.
Li, P., Warner, D., Fatemi, A., Phan, N., 2016. Critical assessment of the fatigue performance of additively manufactured Ti–6Al–4V and perspective for future research. International Journal of Fatigue, 85, 130-143.
Nalla, R. et al., 2002. Influence of microstructure on high-cycle fatigue of Ti-6Al-4V: bimodal vs. lamellar structures. Metallurgical and Materials Transactions A, 33(13), 899-918.
Qiu, C., Adkins, N. J., Attallah, M. M., 2013. Microstructure and tensile properties of selectively laser-melted and of HIPed laser-melted Ti–6Al–4V. Materials Science and Engineering: A, 578, 230-239.
Reeves, P., 2009. Additive Manufacturing – A supply chain wide response to economic uncertainty and environmental sustainability. Econolyst Limited, The Silversmiths, Crown Yard, Wirksworth, Derbyshire, DE4 4ET, UK.
Song, B. et al., 2012. Effects of processing parameters on microstructure and mechanical property of selective laser melted Ti6Al4V. Materials & Design, 35, 120-125.
Spierings, A., Herres, N., Levy, G., 2011. Influence of the particle size distribution on surface quality and mechanical properties in AM steel parts. Rapid Prototyping Journal, 17(3), 195-202.
Thöne, M. et al., 2012. Influence of heat-treatment on Selective Laser Melting products–eg Ti6Al4V. Twenty-Third Annual International Solid Freeform Fabrication (SFF) Symposium.
Vilaro, T., Colin, C., Bartout, J.-D., 2011. As-fabricated and heat-treated microstructures of the Ti-6Al-4V alloy processed by selective laser melting. Metallurgical and Materials Transactions A, 42(10), 3190-3199.
Vrancken, B., Thijs, L., Kruth, J.-P., Van Humbeeck, J., 2012. Heat treatment of Ti6Al4V produced by Selective Laser Melting: Microstructure and mechanical properties. Journal of Alloys and Compounds, 541, 177-185.
Welsch, G., Boyer, R., Collings, E., 1993. Materials properties handbook: titanium alloys. 2 ed. s.l.:ASM international.
Wohlers, T., 2013. Wohlers Report, s.l.: Wohlers Associates.
Wycisk, E., Emmelmann, C., Siddique, S., Walther, F., 2013. High cycle fatigue (HCF) performance of Ti-6Al-4V alloy processed by selective laser melting. Advanced Materials Research, 816, 134-139.