MICROSTRUCTURE AND MECHANICAL PROPERTIES OF DIRECT METAL LASER SINTERED TI-6AL-4V[†]

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ABSTRACT

Direct metal laser sintering (DMLS) is a selective laser melting (SLM) manufacturing process that can produce near net shape parts from metallic powders. A range of materials are suitable for SLM; they include various metals such as titanium, steel, aluminium, and cobalt-chrome alloys. This paper forms part of a research drive that aims to evaluate the material performance of the SLM-manufactured metals. It presents DMLS-produced Ti-6Al-4V, a titanium alloy often used in biomedical and aerospace applications. This paper also studies the effect of several heat treatments on the microstructure and mechanical properties of Ti-6Al-4V processed by SLM. It reports the achievable mechanical properties of the alloy, including quasi-static, crack growth behaviour, density and porosity distribution, and post-processing using various heat-treatment conditions.

OPSOMMING

Direkte metaal-laser-sintering is 'n selektiewe lasersmeltvervaardigingsproses wat naby aan netto-vorm onderdele van metaalpoeiers kan produseer. Verskeie materiale is geskik vir lasersmeltvervaardiging, onder andere titaan, staal, aluminium en kobaltchroom legerings. Die doel van dié navorsing is om die materiaaleienskappe van lasersmeltvervaardigde onderdele te ondersoek. 'n Titaan legering (Ti-6Al-4V) wat dikwels biomediese en ruimte toepassings het, word voorgehou. Verder word die effek van verskeie hittebehandelings op die mikrostruktuur en meganiese eienskappe van die titaan legering, na dit lasersmeltvervaardiging ondergaan het, ondersoek. Die quasi-statiese kraakvoortplanting, digtheid- en poreusheidsverspreiding en die verwerking met verskeie hittebehandelingtoestande word bespreek.

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1   INTRODUCTION

Direct metal laser sintering (DMLS), a selective laser melting (SLM) process, is a laser-based additive manufacturing (AM) technique that uses CAD models to create three-dimensional parts. The technique uses a high-powered laser to fabricate dense components from metal powder [1-3]. DMLS is capable of producing geometrically complex designs to high tolerances and with minimal material waste, while avoiding lengthy machining times. Furthermore, no tooling changes are required for different components to be manufactured on the same machine.

Over the past decade, increased material performance of SLM-manufactured components has allowed for a wide range of applications of the technique [4-6]. One such example is the use of DMLS to fabricate medical implants using Ti-6Al-4V, a titanium alloy that is typically characterised by high strength, low density, high corrosion resistance, and good biocompatibility [7-9]. The use of DMLS has demonstrated its versatility here, as it allows for the manufacture of geometrically complex and customised patient-specific implants. And due to the seamless CAD-to-manufacture transition, fast manufacturing of parts is possible [4].

There is significant concern, however, about the application of SLM-produced parts. For example, medical implants require strict material properties that have not yet been completely matched by SLM products [10]. The concerns abut SLM-manufactured metals relate to internal stresses (resulting from steep temperature gradients and high cooling rates) that occur during the manufacturing process [11], the microstructure of as-built components and the resultant material performance [12], and the occurrence of pores [13]. DMLS parts do not normally have full density (although 99.8 per cent density can be achieved [10,11]), and they have an anisotropy due to the inherent layer-wise building procedure. Furthermore, little has been reported in the literature on heat-treatments, particularly for the application of Ti-6Al-4V in the biomedical industry. According to ASTM F1472 [14], as-built SLM-manufactured Ti-6Al-4V implants are currently may not be used in biomedical implants due to the presence of a martensitic microstructure [12] and the occurrence of porosity [13].

Previous studies by the author have indicated that high, non-uniform residual stresses are present in as-built DMLS Ti-6Al-4V samples that approached the yield strength of the material [11,15]. These stresses could easily be relieved, however, through heat-treatment. Studies by Vrancken et al. [12] on the microstructure and the influence of heat-treatment have shown that, due to the specific process conditions and thus the specific microstructure, SLM-produced parts require different heat-treatment from bulk alloy parts. They showed that the temperature, time, and cooling rate play an important role. Mechanical properties were dependent on the maximum heat treatment temperature where an increased maximum temperature resulted in a decline in the yield and ultimate tensile strength (UTS) and an increase in the fracture strain due to the transformation of the fine α' needles to a more coarse mixture of α and ß. Similarly, work undertaken by Leuders et al. [13] has shown that porosity influences the fatigue life of SLM-manufactured Ti-6Al-4V: they identified a correlation between porosity and the fatigue behaviour in the high-cycle fatigue regime, where porosity vastly decreases fatigue life.

It follows that the successful industrial application of DMLS-manufactured Ti-6Al-4V components requires an investigation of material performance. Such a study should address the achievable mechanical properties of DMLS-manufactured Ti-6Al-4V, and post-treatment – such as heat-treatment – to improve the material performance. Characteristic quasi-static, crack growth and fatigue behaviour, residual stresses, and, most importantly, the microstructural interaction with the properties mentioned, should be investigated.

In this work, DMLS Ti-6Al-4V is post-processed through heat-treatment and hot isostatic pressing (HIP). The material performance is compared with the as-built and the wrought

material condition. A thorough testing procedure, combining porosity investigations using X-ray computed tomography (CT), quasi-static and dynamic mechanical loading, and microstructural characterisation is undertaken. Based on the findings, conclusions are drawn on the applicability of DMLS Ti-6Al-4V for biomedical implants.

2   EXPERIMENTAL METHODOLOGY

Fully characterising the material properties of DMLS-manufactured Ti-6Al-4V and the link between the DMLS process and the material properties requires a vast number of test specimens and test data, and a great deal of time. In this study, the focus was directed toward heat-treatment and the link between microstructure, as well as their link to the achievable tensile and dynamic mechanical properties of DMLS Ti-6Al-4V. This study did not consider any anisotropic affects that arise due to the inherent building process; samples were thus built in the XY plane orientation, according to the ISO/ASTM52921-13 designations.

All samples were made of Ti-6Al-4V, and were produced with an EOSINT M280 (EOS GmbH), which used a layer thickness of 30µm and a 200W Yb-fibre laser. The scanning strategy is multidirectional with no further parameter descriptions supplied by EOS. The machine is installed at the Centre for Rapid Prototyping and Manufacturing at the Central University of Technology in Bloemfontein.

The experimental procedures presented in this study were conducted in ambient conditions. Heat-treatments and density determination (CT scans) were carried out at Stellenbosch University. Tensile tests and investigations in crack growth behaviour were conducted at the Centre for Materials Engineering at the University of Cape Town. Hot isostatic pressing (HIPing) was undertaken by Bodycote in Belgium.

2.1 Heat-treatment and microstructural evaluation

Ti-6Al-4V is an alpha-beta (α-β) alloy that is widely known to be suitable for heat-treatment, with many different microstructures obtainable through variations of heat-treatments. At room temperature, mill-annealed Ti-6Al-4V is about 90 per cent (volume) α, and the α phase thus dominates the physical and mechanical properties of this alloy; the β phase can be manipulated in amount and composition through heat-treatment.

The overall effects of processing history and heat-treatment on microstructure are complex, where the microstructure depends on both processing history and heat-treatment [16]. The microstructure that combines the highest static strength and ductility is not necessarily that which provides the optimum fracture toughness, fatigue strength, or resistance to crack growth. Typically, when wrought Ti-6Al-4V is heat-treated in the α-β temperature range and subsequently cooled, an equiaxed microstructure is formed that is categorised by the presence of globular (equiaxed) primary α in the transformed β (platelike) matrix. Similarly, a β structure is achieved by cooling from above the β transus to obtain an acicular or needle-like structure. The relative advantages of equiaxed and acicular titanium alloy microstructures include a higher ductility and formability, higher strength, and better low-cycle fatigue (initiation) properties for an equiaxed microstructure, as well as superior creep and crack growth properties and higher fracture-toughness values for an acicular grain structure [16].

Previous microstructure studies on SLM-produced Ti-6Al-4V have reported that the as-built material condition has a martensitic microstructure; the matrix is composed of acicular α phase, while no β phase is present [12]. It has also been reported that heat-treatment causes the transformation of the metastable martensite into a biphasic α+β matrix, with a morphology that depends on the heat-treatment [12]. These reported observations compare well with wrought Ti-6Al-4V, and the thought follows that DSLM Ti-6Al-4V can be tailored to achieve the desired mechanical properties that are comparable to wrought Ti-6Al-4V.

In this study, a total of four heat–treatments – including a recrystallisation anneal, duplex anneal, beta anneal, and HIP – were completed. Since Ti-6Al-4V will readily oxidise when heated above 427°C, heat-treatments were undertaken either in a vacuum furnace or with the use of ceramic coatings.

- Recrystallisation anneal (RA): Heated to 950°C, one-hour holding period, followed by a furnace cool.
- Hot isostatic pressing (HIP): heated to 915°C at 1000bar isostatic pressure, two-hour holding period, furnace cooling at 11°C/min.
- Duplex anneal (DA): heated to 950°C, one-hour holding period followed by air cooling, then heated to 700°C, two-hour holding period, followed by air cooling.
- Beta anneal (BA): heated to 1030°C, one-hour holding period followed by air cooling, then heated to 630°C, followed by air cooling.

2.2 Porosity and flaw investigations

The CT scanner is a General Electric Phoenix V|Tome|X L240 with an additional NF180 option. In this study, 5 mm diameter cylinders were scanned allowing for a voxel resolution of 3 μm^3. One cylinder was scanned per post-processing condition.

2.3 Tensile strength and fracture toughness

Characterisation of the quasi-static properties was performed according to ASTM E8/E8M-11. Tests were conducted using the Zwick/Roell 1484 at ambient conditions. The tensile properties investigated were yield strength, ultimate tensile strength (UTS), percentage elongation at break, and Young's modulus. A minimum of five round tensile specimens (with a gauge diameter of 4.00 mm) were tested in the parallel to build direction. All tests were displacement controlled at a strain rate of 0.001/s.

The fracture toughness tests were conducted according to ASTM E399. To reduce the number of samples, fracture toughness tests made use of the samples used for crack growth rate investigations. The fatigue crack length was thus restricted to the range from 0.45 to 0.55 for K_{Ic} determination. Fracture toughness tests were displacement controlled at a rate of 1 mm/min, according to the standard. A minimum of five specimens (with a gauge diameter of 4.00 mm) were tested in the perpendicular to build direction.

2.4 Crack growth rate

An analysis of crack growth behaviour was conducted using compact tension specimens at ambient conditions with a stress intensity factor (K) ratio of R ≈ 0.1. The specimens were manufactured according to ASTM E 647-08 with W = 25 mm and B = 12.5 mm. An optical microscope was used to monitor crack propagation, allowing for crack measurements to be taken every 0.1 mm. Five specimens were tested in the as-built condition, five in the recrystallisation annealed condition, and five in the HIPed condition, totalling 15 specimens. Tests were carried out at a frequency of 10 Hz.

3 EXPERIMENTAL RESULTS

3.1 Density evaluation

The experimental results of the CT scans are presented in Table 1. It shows that a near-full density of 99.79 ±0.2 per cent and 99.94 ±0.2 per cent for as-built and HIPed samples was achieved respectively. These values are in agreement with Knowles et al. [11], who determined a relative density of 99.7 per cent for DMLS Ti-6Al-4V using both Archimedes and optical measuring techniques. Figure 1 shows the CT scan of the as-built and HIPed samples that provide information on the porosity distribution and orientation. Scans in the as-built condition show an even porosity distribution, with no indication of a dependence on the layer distribution or sample orientation.

Table 1: Density of DMLS-manufactured Ti-6Al-4V. Relative density values given to ±0.2%.

	HIPed	as-built
Density [g/cm3]	4,427	4,421
Relative density	99,94%	99,79%

No porosity is visible in the CT scans after the HIP process (Figure 1a), suggesting that any residual porosity is below the CT scan resolution limit of 3 μm. This is in agreement with the work done by Leuders et al. [13], who found a relative density of 100 per cent after the HIP process, measured using CT with a minimum resolution of 22 μm. They found that the mean relative density of as-built samples was 99.77 per cent.

3.2 Microstructure evaluation

Previous studies by Knowles et al. [11] have shown that the as-built microstructure has a very fine acicular (plate-like) morphology. This is attributed to the inherent rapid cooling of the material during DMLS, resulting in a beta-to-martensite transition. XRD analysis by Luca Facchini et al. [17] indicated the presence of only the α phase, which can be recognised as both the α phase and the α' martensite [18-20]. However, because of the very large solidification undercooling, the microstructure may be interpreted as martensitic. This microstructure morphology exhibits a high strength and hardness and low ductility, and can be detrimental to fracture toughness. However, martensite impedes dislocation motion, which leads to a strengthening effect that can improve fatigue crack propagation [16].
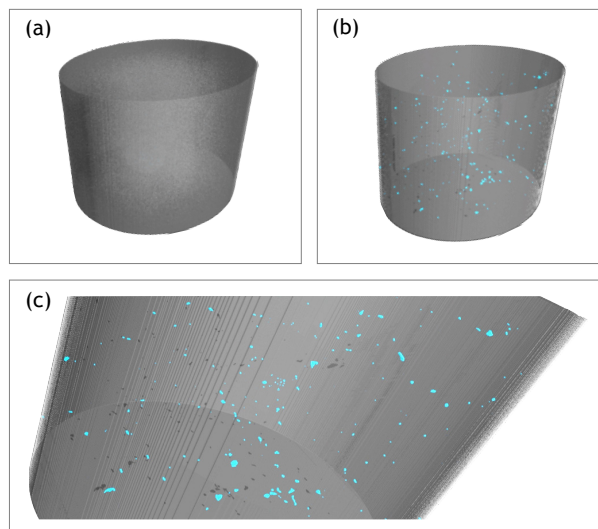

Figure 1: CT scan of a 3 mm diameter cylinder showing porosity (blue coloured) and distribution, where a) HIP, b) as-built. a) After the HIP process the porosity was below the resolution limit of 3 μm. c) is an enlargement of b) to show the pore orientation and distribution.

Figure 2 shows the microstructures obtained through the aforementioned heat-treatment variations. The RA (Figure 2b, 950°C - furnace cooled) shows a microstructure transformed into a Plate-like α + β, consisting of a small amount of equiaxed α. The HIPed condition (Figure 2d, 915°C - cooled at 11°C/min) shows a similar morphology; however, it consists of a larger grain structure and with less equiaxed α, which may be attributed to the faster cooling rate of the HIP treatment. The DA treatment (Figure 2a, 950°C – air cooled, followed by 700°C - furnace cooled) has transformed the microstructure into a matrix of equiaxed and acicular α and a small amount of intergranular β. Lütjering et al. [21] showed that the α colony size is a determining factor for the mechanical properties in wrought Ti-

6Al-4V. The coarsened α phase and larger α colony size result in a reduced tensile strength and an increased ductility, yet do not yield the best crack growth behaviour. Fine grains show a superior long through-crack fatigue crack growth behaviour and fracture toughness [16].
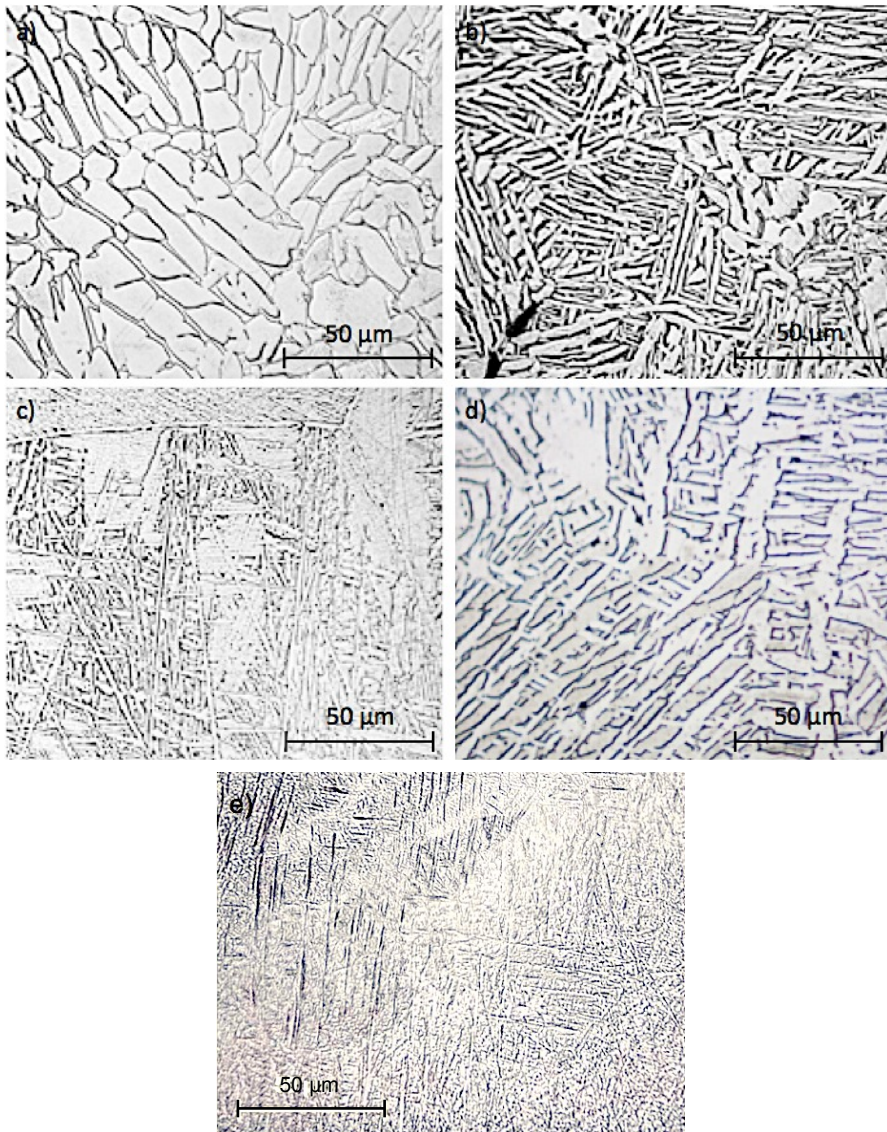

Figure 2: Microstructures obtained through various heat-treatments of DMLS TI-6Al-4V. (a) Duplex anneal (DA), (b) recrystallisation anneal (RA), (c) beta anneal (BA), (d) hot isostatic pressing (HIP), and (e) as-built condition.

The BA also produced changes on the microstructural level, as shown (Figure 2c, 1030°C – air cooled, followed by 630°C – air cooled). The microstructural change produced a fine microstructure consisting of acicular α (transformed β) with the α phase preferentially along the prior- β grains. The 'fineness' of this microstructure is evidence of a moderate cooling rate from above the β-transus. BA microstructures have the lowest fatigue crack growth rates, whereas mill-annealed microstructures yield the highest growth rates [16]. The structure of the Widmanstätten α plus retained β, or a mixture of this structure and α', exhibit yield and tensile strengths superior to those of the mill-annealed wrought metal and a ductility and toughness greater than those of an entirely martensitic microstructure. A summary of the heat-treatments and resultant microstructure is provided in Table 2.

Table 2: Summary of heat-treatments and resultant microstructure

Heat treatment	Holding temperature	Cooling rate	Microstructure
As built	-	-	Fine acicular α' martensite
RA	950°C	Furnace cooled	Plate-like α - β, small amount of equiaxed α
HIP	915°C	11 °C/min	Plate-like α – β, small amount of equiaxed α
DA	960°C and 700°C	Air cooled and furnace cooled	Equiaxed and acicular α with intergranular β.
BA	1030°C and 630°C	Both air cooled	Widmanstätten α + β colony microstructure.

3.3 Tensile and fracture toughness properties

The achievable strengths for the post-treatments are summarised in Table 3. Standard deviations are ±20 MPa for UTS and yield strength respectively, and ±2 per cent for elongation at failure. The as-built samples achieved a high tensile strength of 1155 MPa. The RA, HIP, and DA show a decrease in tensile strength to 914, 960, and 871 MPa respectively. As expected, the BA results in a higher strength of 1008 MPa compared with the below β transis temperature heat-treatments. Similarly, the ductility (per cent elongation) increased through the various heat-treatments, with the DA achieving the highest per cent elongation of 11.5 per cent. These results are consistent with the microstructural observations in that the larger grain structures provided a decrease in strength and an increase in ductility.

The as-built material has a higher strength than the wrought metal, which can be attributed to the fine acicular microstructure. A study undertaken by Vrancken et al. [12] pointed out that, unlike wrought Ti-6Al-4V, the tensile strength decreases and ductility increases with increasing heat-treatment temperature. Wrought Ti-6Al-4V in the mill-annealed condition consists of an equiaxed microstructure; thus a β anneal transforms this microstructure to a lamellar α+β, Widmanstätten α, or martensitic α' microstructure, depending on the cooling rate. The as-built microstructure is in a condition similar to a β anneal; a martensitic α as discussed in Section 3.2. Thus the various heat-treatments outlined – RA, HIP, and DA – essentially anneal a transformed β (which has undergone a severe cooling rate) to a lamellar α+β structure. The outcome of the heat-treatment primarily depends on the cooling rate (unless two or more heat-treatment cycles are undertaken, such as the DA or BA). It would be expected that, based on the understanding of the heat-treatment of wrought Ti-6Al-4V, the thickness of the grain boundary α is dependent on the cooling rate – decreasing with increasing cooling rate – and, when cooled from above the β transus, i.e. the as-built material state, the cooling rate determines the size of the α colonies and decomposition of martensite.

The fracture toughness results are also presented in Table 3. The as-built material condition resulted in surprisingly low toughness of K_{Ic} = 37.5 MPa√m. The HIP condition results in a stress intensity factor of K_{Ic} = 57.8 MPa√m, and the RA has the highest toughness of K_{Ic} = 86.3 MPa√m. These results are within 5 MPa√m.

The poor fracture toughness in the as-built condition may be explained by the martensitic microstructure and high UTS. Studies on the wrought material have shown that in a superior β transformed microstructure, an increase in UTS significantly decreased the toughness of the material, where an increase of about 20 per cent in UTS can halve K_{Ic} [16]. The low HIP fracture toughness is somewhat surprising when comparing the result with the RA toughness. It is not clear why the HIPed condition would result in a significantly lower toughness; it could, however, be attributed to a larger grain size.

Table 3: Ultimate tensile strength, elongation at failure and fracture toughness of Ti-6Al-4V. Wrought and P/M (powered metallurgy) results were taken from Donachie [16].

Process and heat-treatment		UTS [MPa]	% elongation at break	K_{Ic} [MPa$\sqrt{}$m]
DMLS	as-built	1155	4.1	37
Wrought	Mill-annealed	930-970	17-19	44-66
DMLS	Stress relief	1230	7.0	-
DMLS	Recrystallisation anneal	914	10.3	86[†]
DMLS	Duplex anneal	871	11.5	-
DMLS	Beta anneal	1008	8.4	-
Wrought	Beta anneal	875-915	-	88-110
DMLS	HIPed	960	8.5	58
P/M	HIPed	937	17	85

3.4 Crack growth behaviour

The results obtained from crack growth measurements are shown in Figure 3. The data is compared with the results obtained by Leuders et al. [13], and shows excellent agreement (Leuders' data is based on samples built on a SLM 250HL machine from SLM Solutions GmbH). The data shows a considerable difference between the as-built and heat-treated material condition. The as-built condition has considerably poorer fatigue performance relative to the heat-treated condition (identified by the faster crack growth rate for a similar cyclic stress intensity factor value), and exhibits large scatter. Specimens in both the RA and the HIP condition have significantly less scatter and a similar behaviour to that of the wrought mill-annealed metal. The scatter in data may be attributed to high residual stresses that are present in the as-built state.

Similarly to the quasi-static properties, the processing and microstructure can have a large effect on the crack growth rate. Generally for wrought metal, BAed microstructures in near-α and α - ß have the lowest fatigue crack growth rates, whereas mill-annealed microstructures yield the highest growth rates. A martensitic α microstructure can be advantageous; however, in the case of DMLS, the high residual stresses probably outweigh any advantageous microstructural effects. A recent study by Rafi et al. [22] showed an improved fatigue performance for as-built SLM samples compared with annealed cast samples. It is not clear in their study, however, whether any stress relieving was performed.

Furthermore, the HIPed samples show little, if any, improvement in fatigue crack growth. However, the work done by Leuders et al. [13] showed that for mean stress-based fatigue life investigations, the fatigue failures ranged from $27 \cdot 10^3$ to $290 \cdot 10^3$ cycles to failure for as-built and heat-treated samples. By contrast, none of the HIPed specimens failed during tests, which were interrupted at $2 \cdot 10^6$ cycles at a cyclic stress of 600 MPa. Clearly there is a strong interaction between fatigue life and porosity. Pores act as sites of increased stress, and thus act as crack initiation sites.

4   DISCUSSION

Ti-6Al-4V is one of the most widely-used titanium alloys, with an excellent combination of strength and toughness along with excellent corrosion resistance. Relying on the refinement of the grains upon cooling from the ß region or the α-plus-ß region develops the properties of this alloy, and subsequent low-temperature aging allows the martensite formed upon quenching to decompose.

Clearly, a significant improvement in the material performance of DMLS Ti-6Al-4V can be achieved using an appropriate treatment. Both ductility and toughness, as well as the crack growth characteristics, may be substantially improved. Although HIPing does not have a

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[†] $P_{max}/P_Q \leq 1.1$ was not achieved.

direct effect on these properties, Leuders et al. [13] have shown that non-HIPed samples have a very poor fatigue life, where pores act as crack initiation sites.
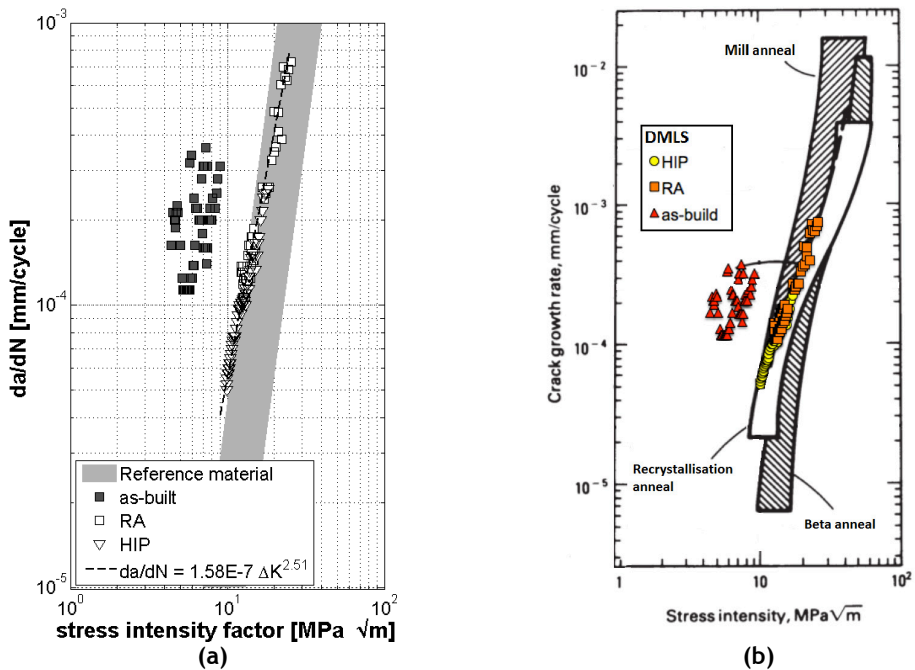

Figure 3: Crack growth curves for DMLS Ti-6Al-4V. (a) DMLS data in comparison with SLM-produced TI-6Al-4V [13] (reference material). (b) DMLS data superimposed on to Ti-6Al-4V production methods data (data reproduced from Donachie [16]).

To obtain high strength with adequate ductility, one heat-treatment below the β transus is suggested. If higher fracture toughness is required, BA may be desirable. However, heat-treating Ti-6Al-4V in the β range can cause loss in ductility. As is typical for wrought Ti-6Al-4V, a heat-treatment just below the β transus can obtain an optimum balance of ductility, fracture toughness, and tensile strength.

It is important to realise that standard heat-treatments do not apply to DMLS material. In Ti-6Al-4V, the microstructure, and hence the mechanical properties, depend on the process history. Since as-built DMLS Ti-6Al-4V has a fine acicular morphology that results from the heating above the β transus, improper heat-treatment can result in undesirable properties. For example, α phase can form preferentially along the prior-β grains. Because cracks tend to propagate in or near interfaces, this type of structure can provide loci for crack initiation and propagation, and thus lead to premature failure. Correct BA microstructures have the lowest fatigue crack growth rates, whereas mill-annealed microstructures yield the highest growth rates (Figure 3b).

5   CONCLUSIONS

The aim of this study was to evaluate the achievable material performance of DMLS Ti-6Al-4V through heat-treatment. In order to establish a link between mechanical properties and heat-treatment, a comparison of the various heat-treated microstructures (and porosity) was undertaken. The findings presented in this study lead to the following conclusions:

- The tensile behaviour, fracture toughness, and crack growth behaviour compare well with the wrought material. As for most commercial SLM processes, a nearly-100 per cent dense material may be achieved.
- The material behaviour is directly related to its microstructure, which can be tailored by specific heat-treatments. The as-built condition has an unfavourable performance

9

that is strongly inhibited by large residual stresses with an unfavourable martensitic microstructure.

- Due to the specific process conditions and hence microstructure, DMLS-produced parts may require heat-treatment that is different from the wrought material.

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