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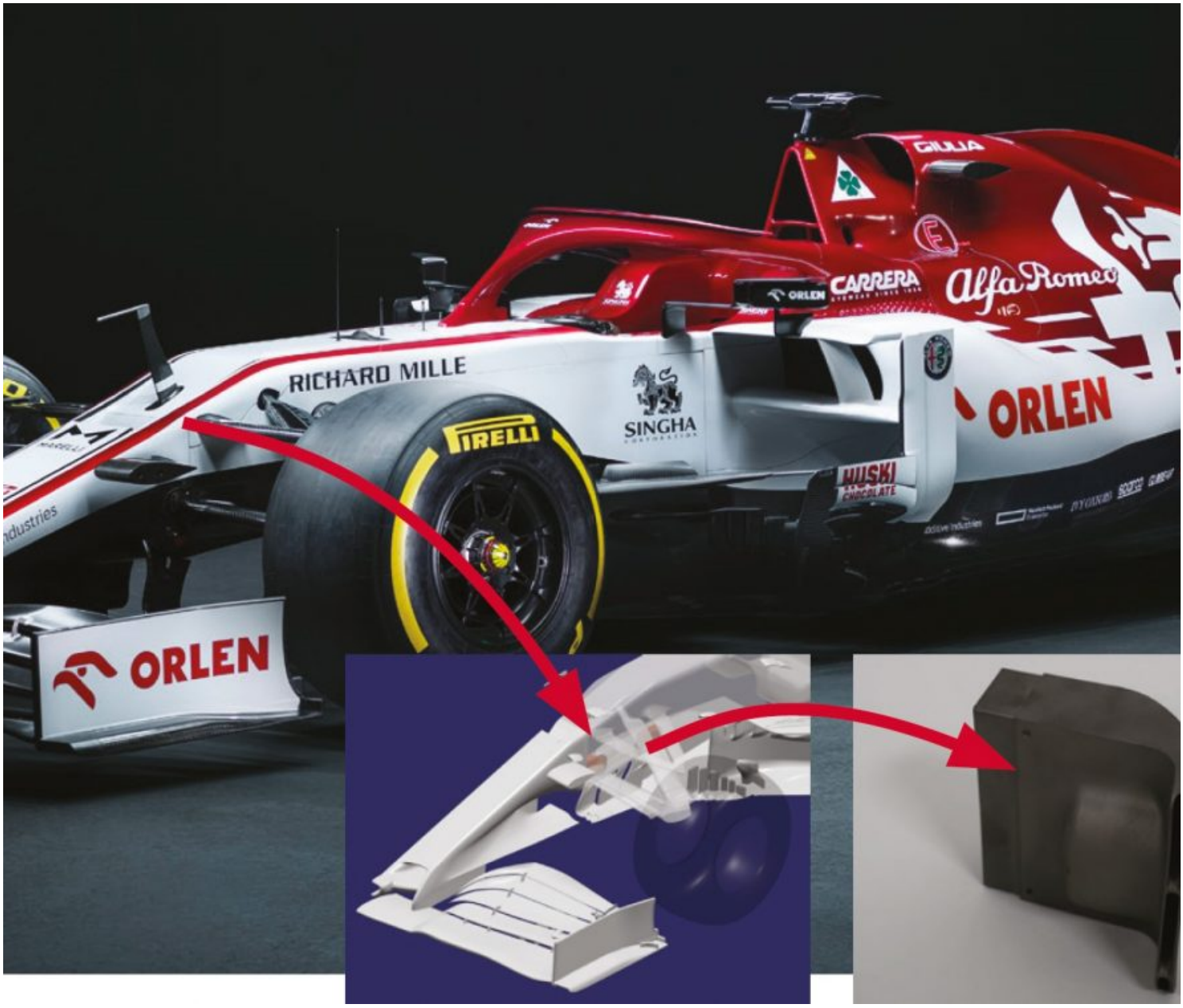


January 22nd, 2021

The game changer in additive manufacturing - Hiping of additive manufactured Titanium components

Optimized HIP and Heat Treatment for Fatigue Strength of Additively Manufactured Ti6Al4V

1. Introduction

Critical components made of high-performance materials subjected to fatigue loads have extreme demands on qualification and production control. Additive Manufacturing is now an increasingly important technology for implementation of bionic lightweight designs.

Over recent years, a great deal of research work has been carried out on additively manufactured grade 5/23 titanium (Ti6Al4V) in safety-critical applications such as aerospace, medical or car racing. Lightweight designs bring about lower safety margins, which in turn create a need for high fatigue strength together with a robust production process resulting in a very low standard deviation of mechanical material properties.

Post-processing, mainly consisting of hot-isostatic pressing (HIP), heat treatment (HT) and surface finishing, is an integral part of a robust production process and should be ideally tailored to a particular alloy. This is especially interesting and rewarding in the case of such a widely-used and well-researched alloy as Ti6Al4V, taking the best out of both the novel microstructure of the AM process as well as of more established heat treatment methods known from forgings or castings.

High-pressure Heat Treatment (HPHT), developed by Quintus Technologies for combining traditional HIP cycles with an integrated, subsequent inert gas quenching cycle is used with great advantage on additively manufactured Ti6Al4V for maximizing the fatigue strength in a lean, fast and robust process.

2. The Healing of Defects by HIP for Enhancing HCF Strength

The mechanical properties, especially fatigue resistance, of additively manufactured components in their as-built condition are generally lower compared to components produced using conventional methods such as forging. The process of adding layer after layer gives rise to pores, pockets of unfused powder, micro cracks and residual stresses. These defects cause stress concentration and act as crack initiation points, having a detrimental effect on fatigue resistance and ductility.

The Kitagawa diagram (Figure 2) shows for a given defect size (equivalent crack length) the allowable stress amplitude for infinite life, with the “fail” and “no-fail” areas above and below the curve. The impact of microscopic defects in the material on the fatigue limit of a fail-safe component is evident.

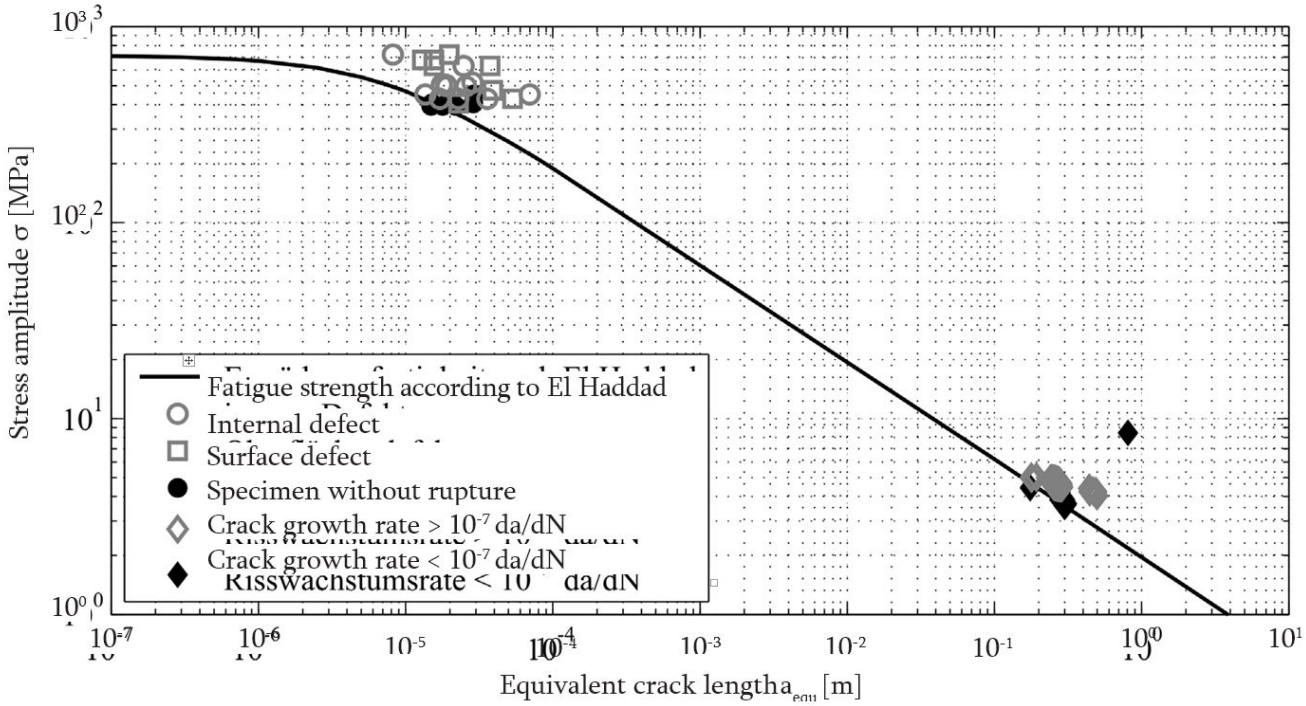


Figure 2. Kitagawa-diagram [1] of Ti6Al4V material produced by L-PBF. Based on the experimental data, a theoretical maximum stress amplitude of 790MPa was determined for a defect-free L-PBF microstructure.

Albeit different notch geometry and stress intensity factors compared to internal material defects, surface defects and surface roughness also strongly reduce the allowable stress amplitude. For L-PBF Ti6Al4V, M. Benedetti et.al. [2] have found that HIP (for curing of internal material defects) in combination with shot peening for surface modification resulted in the best improvement of fatigue strength. This can be attributed to the fact that shot peening not only smooths the surface but also transforms the residual tensile stresses from the build process – which can be has high as 400MPa [3] – into surface compressive stresses.

The successful use of HIP for elimination of internal material defects and increase of the fatigue strength of L-PBF Ti6Al4V has been studied and confirmed by many authors in recent years, for example [4]. The widely used HIP parameters for L-PBF Ti6Al4V is 920°C, 100 MPa and 2 hours hold time, thus within the specifications of ASTM F2924-14, will reliably increase the allowable stress amplitude at N=106 dramatically from less than 400 to more than 600MPa (see Figure 3).

These process parameters were originally developed and intended for HIP densification of pores and shrinkages in titanium castings, which solidify normally in a microstructure closer to equilibrium than is the case for L-PBF material, which shows a more pronounced softening at elevated temperature treatment than castings.

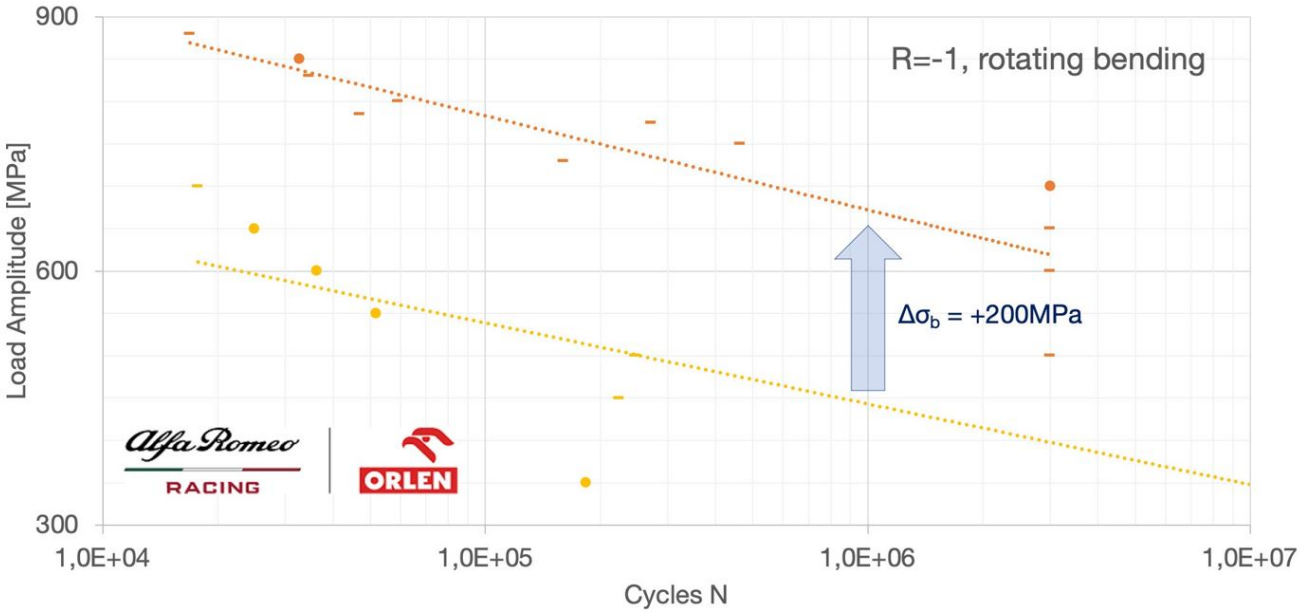


Figure 3. Rotating bending fatigue strength of L-PBF Ti6Al4V in the stress-relieved (SR, 720°C) and SR+HIPed conditions (by courtesy of Alfa Romeo Racing ORLEN ©2020)

3. The Softening of Ti6Al4V at Elevated Temperature

It has been reported that Ti6Al4V material produced by L-PBF is showing a noticeable decrease of static strength with any kind of elevated temperature treatment [4]. This is attributed to the metastable state of the extremely rapidly cooled microstructure in the “as-built” condition. In-situ cooling rates of 104-106 K/s are reported [5], leading to a complete martensitic transformation into the α’ phase. Even though some authors are reporting the Mf temperature of Ti6Al4V below room temperature, no presence of retained β was confirmed for example in the XRD experiments by J. Mezzetta [6] in the “as-built” condition (Figure 4).

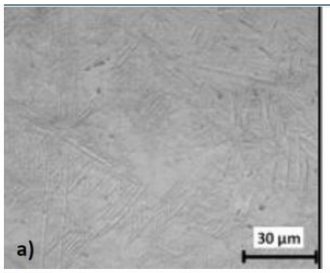
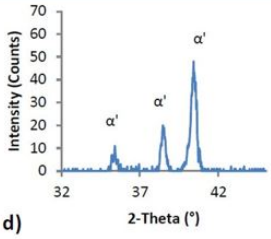
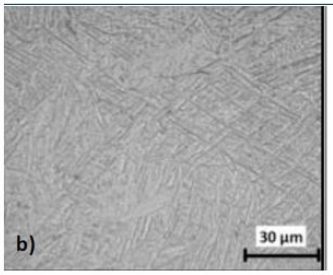
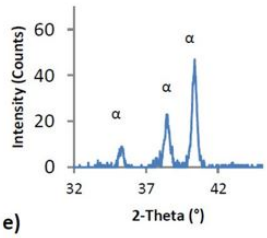
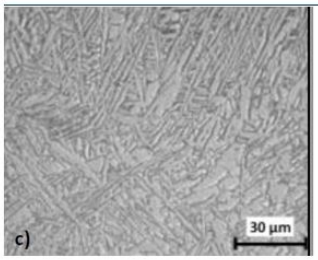
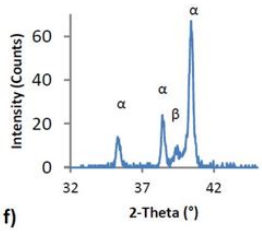
Micrographs and XRD spectra	Heat treatment condition	α/α' platelet width
 	SR (stress relief), acc. to AMS2801 (593°C) no decomposition of α'	1.0±0.6 µm
 	ANN (anneal), acc. to AMS2801 (704°C) α' –> α	2.0±0.8 µm
 	HIP (hot isostatically pressed), acc. to ASTM F2924 (895-955°C) α' –> α + β	3.2±0.6 µm

Figure 4. Progressive change of microstructure (α/α' platelet width) and martensite phase decomposition (α'-> α+β) with increasing heat treatment temperature (adopted from [6]).

The dependence in fully lamellar microstructures of features such as α-colony size and α-platelet width of the cooling rate and their importance for strength (Rp0.2, UTS), ductility (εF) and HCF-strength has been emphasized for example by G. Lütjering [7], and he describes the onset of martensitic phase decomposition at 700 – 850°C. This is associated with usually reported [8] losses of yield strength from the as-built condition:

- from approx. 1050 to 950 MPa during elevated temperature stress relief (= mill annealing ANN acc. to AMS 2801) at 700-800°C
- and from approx. 1050 to 900 MPa during elevated temperature HIP at >900°C (acc. to ASTM F2924-14, where complete martensite decomposition will)

4. Low-temperature HIP to Minimize Softening

Together with partners [8,9], Quintus Technologies has developed, evaluated and implemented a new low-temperature HIP cycle optimized for the very fine "as-built" L-PBF Ti6Al4V microstructure. The underlying concept is to minimize the elevated temperature softening by lowering the HIP temperature as much as possible and compensate the temperature reduction by an increase of the pressure.

Excluding superplastic deformation as a high-temperature deformation mechanism, which only occurs in equiaxed microstructures of Ti6Al4V, three deformation mechanisms occur during HIP of metals [10], namely:

- plastic deformation (dislocation glide), dominant in the low temperature range
- dislocation creep (Norton creep), dominant in the middle temperature range
- diffusion creep (Nabarro-Herring creep), dominant in the high temperature range

For the simplification of HIP parameter optimization, most authors [11] are modelling only the Norton creep. For aluminium alloys Y. Skrinsky [12] has shown in the relevant HIP temperature range of 450- 550°C and relevant pressure range of 100-200MPa, that for a given defect size (=required HIP soaking/ densification time), a 1% increase of the temperature will reduce the HIP-time by 15%, whereas a 1% increase of the HIP-pressure will only lead to a 2.5% reduction of the HIP-time, thus a 6times higher differential impact of temperature.

Q. Xu [13] has used a Norton-creep model for comparing defect densification by HIP at 150MPa in the temperature range of 750 – 950°C with cast samples of Ti6Al4V with lamellar microstructure. He found that casting pores of Ø2.5mm will only completely disappear above 920°C, but that up to 300µm defects will disappear completely after 2hours at 800°C.

This corresponds very well with results obtained with the low-temperature HIP-cycle proposed by Quintus Technologies for L-PBF Ti6Al4V, where the HIP temperature is reduced from 920°C to 800°C and the HIP pressure increased from 100 to 200MPa. Since the mean pore size of the L-PBF micro- structure is one order of magnitude less, typically <100µm, than that of castings, full pore densification is achieved by low-temperature HIP.

T. Kosonen [8], confirmed that this new, low-temperature HIP process will produce approx. the same yield strength as a high-temperature stress relief treatment at 800°C (950MPa) but combining it with defect densification and the attendant HCF resistance. Testing at a stress amplitude of 750MPa, it was found in [1] that with a low-temperature HIP process at 820°C, the theoretical maximum for the HCF resistance of L-PBF Ti6Al4V at N=107 of 790MPa was achieved by all but a narrow margin.

If fracture elongations <12% are allowed, the yield strength can be pushed to even higher levels of 1000MPa with this method, using maximum oxygen contents (2000µg/g).

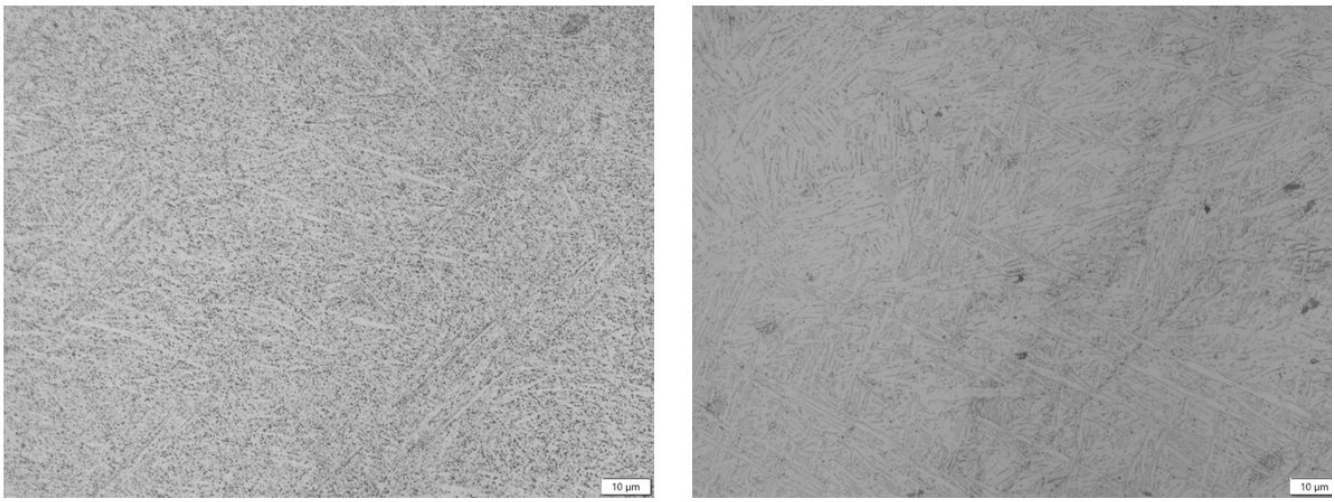


Figure 5a. As-built microstructure of L-PBF Ti6Al4V, showing almost 100% acicular α' martensite with some nano-scale retained β
(adopted from [1])

Figure 5b. Microstructure after low-temperature HIP process at 820°C, showing a decomposition of α' into very fine α platelets and α+β

Although lacking experimental evidence, it can be interpreted from the comparison of figures 5a and 5b that the pressure increase from 100 to 200MPa not only compensates the temperature decrease from 920 to 800°C in order to achieve complete pore densification, but – according to Le Chatelier's principle – stabilizes a little bit more the hcp α-phase with the lower specific volume than the bcc β-phase, thus helping to retain a fine lamellar microstructure during the HIP process.

5. High-pressure Heat Treatment for Maximum Strength

There are cases where a low HIP temperature cannot be applied successfully, for example when the typical or maximum defect size is larger than 300µm [13]. This may not only be the case in castings, but also for example when using so-called "high-speed" build parameters. D. Herzog has found that when accelerating the planar build rate, there is a significant drop of "as-built" density when the volume energy is reduced by a factor of more than 1.7, associated with a jump of the size of defects from <100µm to several hundred µm [14], which were not completely densified using the low- temperature HIP process.

For such applications, Quintus Technologies has developed high-pressure heat treatment (HPHT), which is essentially the provision of a high-pressure gas quenching (HPGQ) device in a HIP furnace. The difference between HPHT and conventional HPGQ lies in the density, velocity, heat capacity and thermal conductivity of the Ar cooling gas at approx. 1500bar versus approx. 15bar. Simplifying the underlying correlations (for example the Wakao or Gnielinski correlations) of the convective thermal transfer coefficient α with the Prandtl and Reynolds numbers of the cooling gas, it can be said that the cooling gas density is approx. 100 times higher in HPHT compared to HPGQ whereas it is the opposite for the gas velocity (of the forced convection cooling gas flow).

This results in similar gas quenching rates in the order of >1000 K/min and thermal transfer coef- ficients of α >500 W/(m2K) in a 1500bar HPHT furnace and a 15bar HPGQ furnace [15] which makes a simultaneous HIP and STA (solution treat and age) heat treatment of Ti6Al4V possible. For Ti6Al4V AMS2801 specifies water or polymer quenching for STA, and this corresponds well with the findings of A. Rottstegge who found that the quench intensity of HPHT lies between oil and water quenching [15].

The HPHT furnace log in Figure 6b shows a typical HPHT (HIP+STA) heat treatment of Ti6Al4V. In this case HIP+ST was carried out at 950°C followed by a 20h ageing at 500°C. The attendant gas quenching rate was 170 K/s shown in Figure 6a. According to J. Sieniawski [16] the minimum cooling rate for martensite transformation is ≥18K/s. Modelling of the convective heat transfer between the cooling gas and the furnace load done at Quintus Technologies is suggesting that the HPHT process is suitable for a HIP+STA treatment of Ti6Al4V and can substitute a conventional high-temperature HIP and sub-sequent water or polymer STA treatment up to 15mm cross sections.

This is industrially relevant, in particular when taking into account that additive manufactured light- weight parts do usually not have thick cross sections and are preferably not quenched in water or polymer in order to avoid formation of α-case.

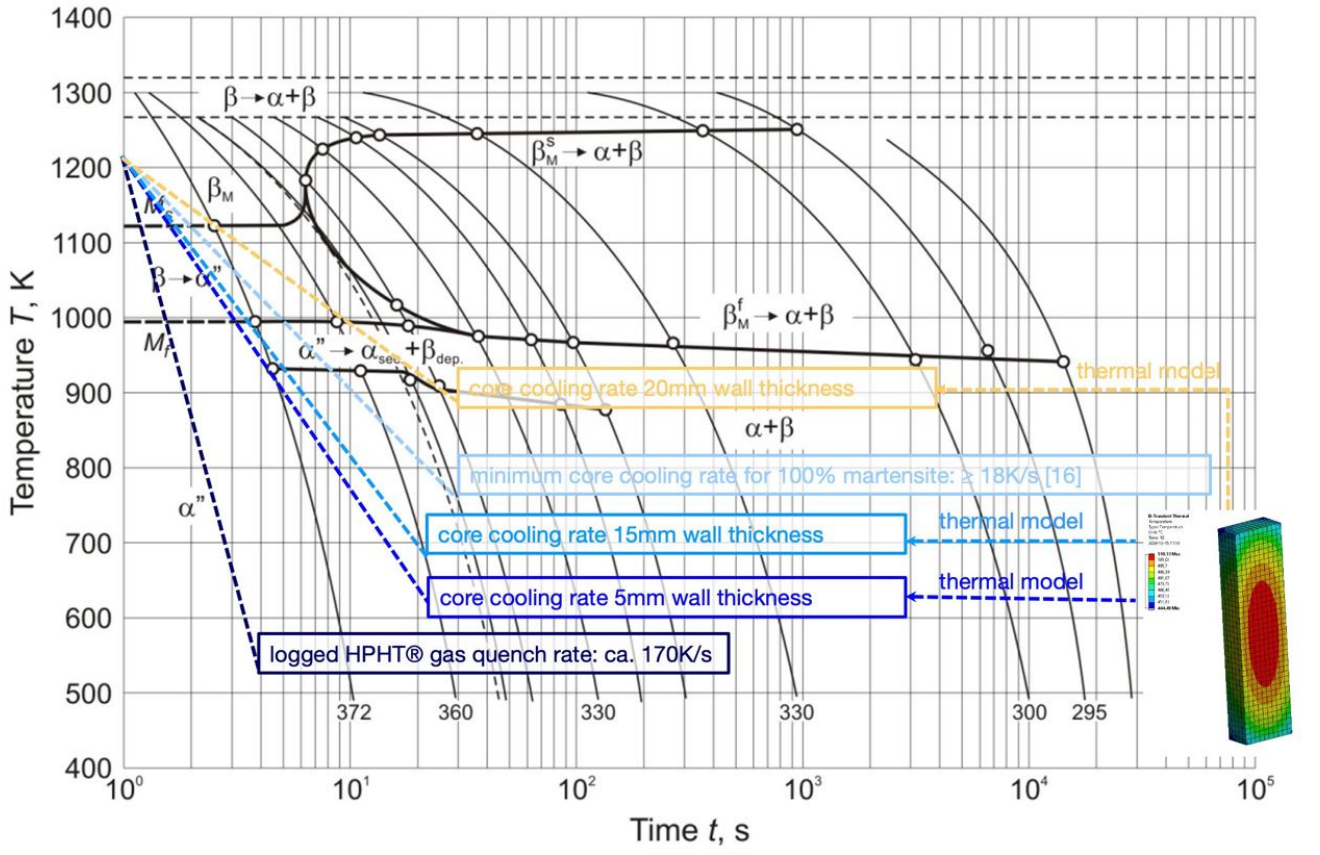
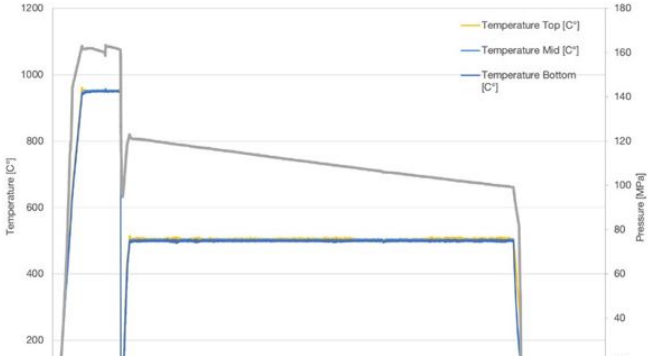
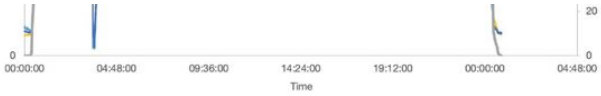


Figure 6a. CCT diagram of Ti6Al4V (adopted from [16]), with overlaid logged furnace gas quench rate of Figure 6b, minimum core cooling rate for martensite transformation and modelled material core cooling rates of 5, 15 and 20mm cross sections.

Whereas AMS2801 specifies only one set of STA parameters which is 941°C ST followed by 482°C/8h ageing, Quintus Technologies have found during several test series with L-PBF Ti6Al4V material that a good combination of strength and ductility can be achieved with 900°C ST followed by 500°C/20h ageing.





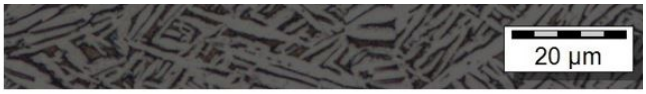


Figure 6b. Furnace log (gas temperature and pressure) during the HPHT (HIP+STA) cycle of the L-PBF Ti6Al4V RBT samples of Figure 3 (solution-treated at 950°C)

Figure 7. L-PBF Ti6Al4V microstructure from the HPHT (HIP+STA) process (adopted from [17], solution treated at 900°C). The primary α-platelets coarsened to approx. 3µm width which is consistent with Figure 4. Not recognizable in the optical micrograph are nanoscale
α+β and α2

Figure 7. shows a microstructure obtained by O. Stelling with these parameters [17]. The corresponding tensile samples exhibited a yield strength of 985MPa in combination with a fracture elongation of 14%.

The HPHT (HIP+STA) process allows to compensate the softening of the material in the normal HIP temperature range of 895-955°C (see chapter 3) due to martensite decomposition (α'–>α) and coarsening of primary α platelets by two counteracting hardening mechanisms:

1. The fast cooling rate leads to transformation of β which has formed above 880°C into α'/α" martensite and a nano-scale bi-lamellar α+β microstructure rather than a softer equilibrium microstructure.
2. The precipitation of coherent α2 (Ti3Al) particles during the ageing

Besides the severity of the quenching cycle, two main influencing factors about the trade-off between maximum strength and ductility, are suggested:

1. Oxygen at the upper specification limit (usually 2000 µg/g) or above will lead to a higher solute drag as well as to a more reliable α2 precipitation [18]. (Consequently, α2 precipitation hardening will not – or only to a very small extent – occur in grade 23 titanium.)
2. Increasing the ST temperature from 900 to 950°C will lead to a higher α' martensite volume fraction in the quenched state due to a higher β volume fraction at 950°C compared to 900°C, as well as to a higher α'/α" ratio, where the orthorhombic α" is the softer martensite structure formed due to a lower vanadium content in β at 950°C compared to 900°C [19].

Varying above factors typically leads to a range of Rp0.2/εF characteristics from 980MPa/14% to 1050MPa/8%, providing room for designers and material engineers to optimize the HPHT (HIP+STA) of L-PBF Ti6Al4V for a given part and application.

6. Case Study: HPHT for L-PBF Ti6Al4V Formula 1 Racing Car Chassis Inserts

About a dozen inserts made of Ti6Al4V are laminated into the CFP sandwich chassis of formula 1 racing cars at various force transmission points (Figure 8). In the front section they transmit all forces, torques, vibrations and shocks of the track and suspension arms as well as all aerodynamic forces from the front spoiler. They also transmit front crash forces from the front crash structure into the chassis.

In the chassis rear section, they are pivotal for the longitudinal and transversal stiffness of the car, transmitting all connection forces, torques, vibrations and shocks between the chassis and the engine mounts.

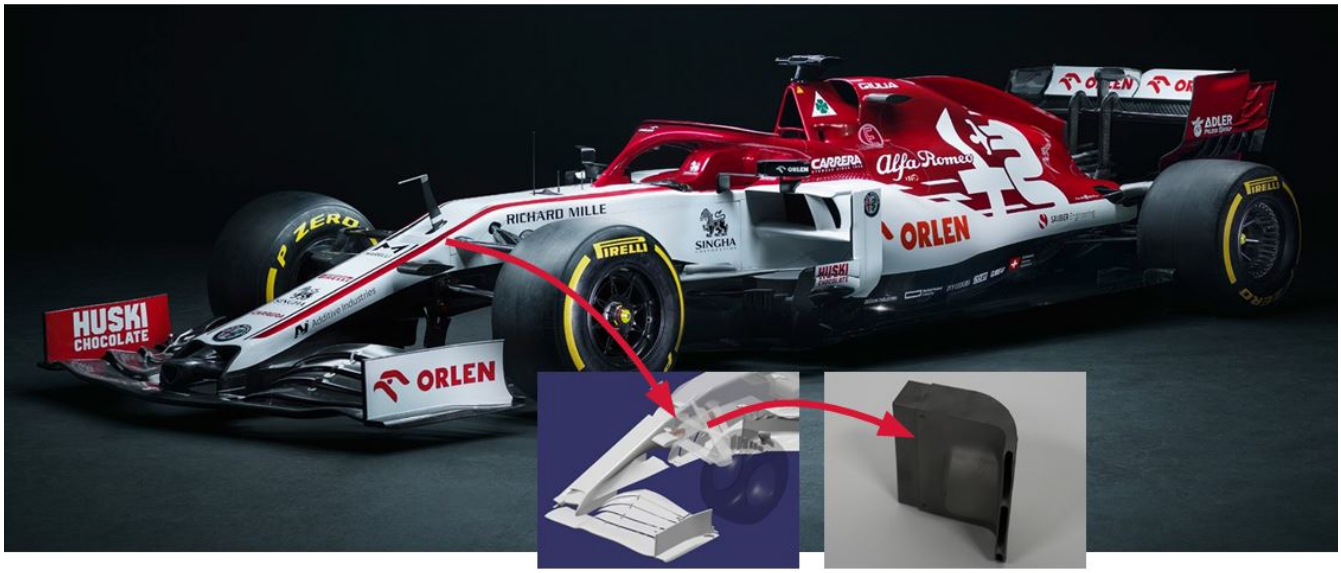


Figure 8. Front chassis inserts (2+2) made of L-PBF Ti6Al4V and post-processed by HPHT (HIP+STA) in C39 formula 1 racing car, Alfa Romeo Racing ORLEN ©2020

Re-engineering the Ti6Al4V chassis inserts for L-PBF by Sauber-Engineering lead to several hundred grams weight savings per insert, which has a huge influence in the performance of the car. The inserts have a very thin cross section in the order of 1mm resulting from the extreme lightweight design, which makes them very suitable for additive manufacturing but also vulnerable to material defects.

Consequently, and for maximizing both strength and fatigue resistance, their surface is conditioned by glass bead shot peening after a HPHT (HIP+STA). Figure 3 provides fatigue strength data and Figures 9a, 9b provide a comparison of the defect indication by NDT/CT before and after HPHT.

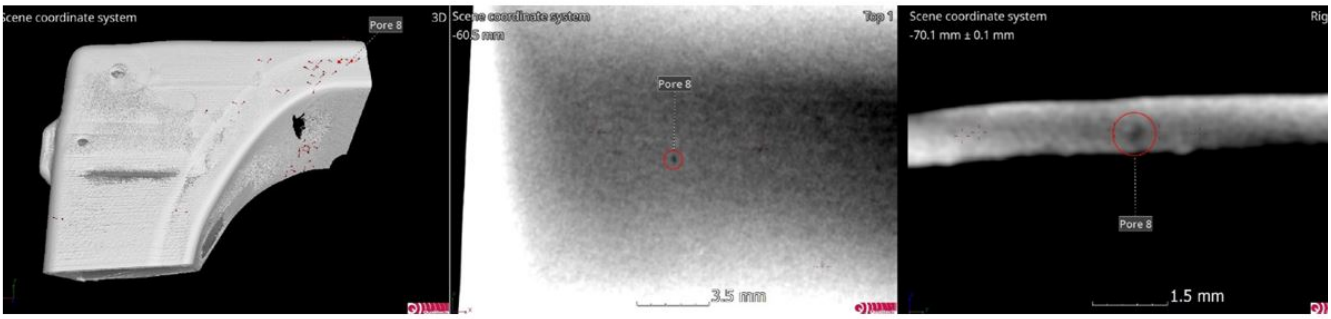


Figure 9a. CT defect indication of “Pore 8” before HPHT (HIP+STA) in Ti6Al4V L-PBF front chassis insert

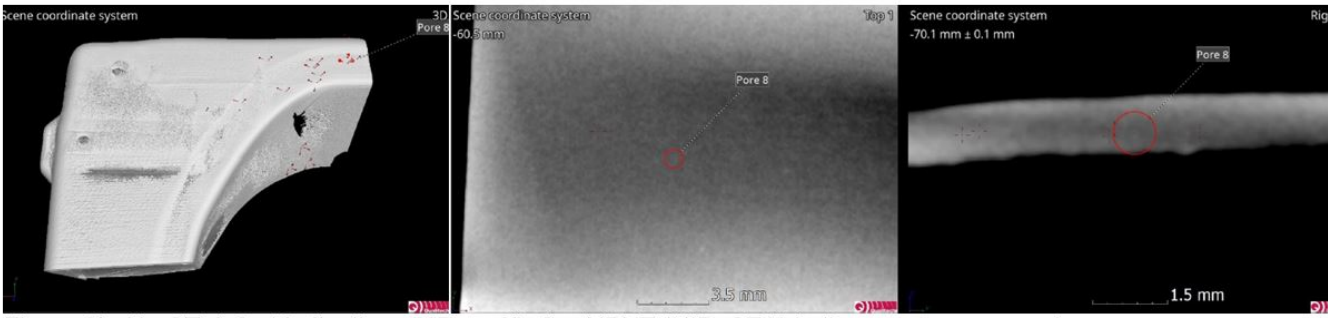


Figure 9b. No CT defect indication of “Pore 8” after HPHT (HIP+STA) in the same component

The designers specify a relatively modest fracture elongation (εF < 7%) which is why the chassis inserts are heat-treated by HPHT (HIP+STA) to a yield strength Rp0.2 well above 950 MPa.

Interestingly, these chassis inserts are highlighting both the vulnerability of lightweight designed AM parts but also their good AM manufacture feasibility (Table 1).

Lightweight design vulnerability	L-PBF	HIP/heat treatment	NDT
Thin cross section	• Higher impact of surface roughness	• Surface-connected pores or microcracks are not densified during HIP	
High static and dynamic loads	• Surface treatment required • Meticulous monitoring of CPPs (critical process parameters)	• Low defect tolerance (surface roughness and volumetric defects), HIP required • Advanced material engineering and heat treatment processes required for maximizing strength	• Meticulous test and inspection planning required

Lightweight design manufacture feasibility	L-PBF	HIP/heat treatment	NDT
Thin cross section	• Relatively compliant structure, less residual stresses • Relatively low SAD (sliced area distribution), low total heat input into powder bed • Fast build time • Relatively low support fraction of SAD	• More choice of heat treatment processes • Robust quenching results • Core quenching rate not so dependent on material conductivity • Stable microstructure throughout cross section • Low thermo-elastic stresses during quenching • Very good results with HPHT (HIP+STA)	• Relatively lower RT (CT) defect size detection limit 
High strength and fatigue strength	• Ti6Al4V L-PBF material equivalent or superior to forged material	• Advanced material engineering and heat treatment processes available and applicable for maximizing strength	

Table 1. Ti6Al4V L-PBF chassis insert lightweight design vulnerability and AM manufacture feasibility

7. Summary

1. Conventional HIP of L-PBF Ti6Al4V acc. to ASTM F2924-14 effectively enables a HCF strength of 600MPa (R=-1, N=106) by densification of typical defects resulting from the build
2. The elevated temperature softening of L-PBF Ti6Al4V associated with conventional HIP tempera- tures of 895-955°C can be avoided by a low-temperature HIP process at 800°C/200MPa. Such material exhibits a HCF strength of up to 750MPa (R=-1, N=107)and retains the same yield strength as high-temperature stress-relieved material (R2 > 950MPa). This process can be used up to a defect size of 300µm.

3. For densification of larger pores of 1-2mm – which can occur with L-PBF high-speed building parameters – and for maximization of yield strength (R2 > 1000MPa), an advanced high-pressure heat treatment (HPHT = HIP+STA) can be successfully applied to high-performance AM parts.

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