Influence of hot isostatic pressing on mechanical response of as-built SLM titanium alloy

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Abstract. Hot Isostatic Pressing (HIP) can be used to increase the fatigue limit of additively manufactured metals. However, with no further surface treatment, its contribution might be restricted by a rough surface. We thus tested Ti6Al4V alloy prepared by selective laser melting in the as-built surface condition to evaluate the utility of HIP. The influence of HIP on microstructure, static mechanical properties and fatigue was evaluated in comparison with untreated and conventionally heat treated state.

1. Introduction

Selective laser melting (SLM) belongs into the group of powder-bed additive manufacture. A powder bed is selectively exposed to a laser beam. The laser beam is moved by a scanning system according to the 2D section of the produced part corresponding to the layer being processed. The powder is locally melted and original particles fuse into a solid part. Further layers of powder are gradually deposited and processed until the product is complete [1].

In general, the properties of components made by means of SLM are mainly determined by the parameters of the SLM process [2]. Material density is one of the first characteristics that are investigated in the search for an optimal SLM processing windows. Varying scanning speed and laser power, for example, changes the dimensions of melt pools, leading to the generation of different kinds of defects such as spherical keyhole pores or crack-like voids due to lack of fusion. Such defects can significantly influence mechanical response of SLM parts, especially under fatigue loading [3]. But even with optimized parameters, completely dense parts cannot be obtained [4].

To enhance the density and improve the properties of SLM parts, hot isostatic pressing (HIP) is increasingly applied for post-processing after SLM. HIP is a technique used commonly for sintering of powders, diffusion bonding or densification of castings. It involves the simultaneous application of a high pressure of a gas and high temperature, which leads to diffusion and plastic deformation [5]. However, it has been shown that the effectiveness of HIP depends on the type of pores. As the SLM chamber is filled with an inert gas, pores formed by gas encapsulation can be formed in final SLM parts. The complete closure of such pores by HIP is hard to achieve. Also, if the porosity is not closed, pressure equalization prevents pores from closing [6].

In numerous studies, HIP was used to improve the fatigue of SLM parts [7-9]. Haan et al [7] showed extraordinary high values of fatigue limit for a Co-based alloy F75 prepared by combination of SLM+HIP. Such improvement was attributed not only to the closure of porosity but also to complete recrystallization. Leuders et al [8] showed a significant improvement in fatigue life of Ti6Al4V samples.
Yu et al [10] studied the effect of post-processing in a broader scale, looking at the effect of surface finishing and heat treatment including HIP. All these post-processings were contributory, however, a vital role in fatigue performance was played by surface condition. With samples polished to Ra = 0.04 µm and HIPed, the highest fatigue limit of 450-500 MPa was reached. Similarly, Vayssette et al [11] showed an increase from 222.5 to 512.5 MPa after machining HIPed samples of Ti6Al4V alloy.

Although there were several papers dealing specifically with HIP of SLM Ti6Al4V alloy [3,8,10,12], not many among them discussed microstructural changes associated with elevated temperature of HIP and their share in changing mechanical properties. Moreover, fatigue tests were mostly performed on machined samples. Nevertheless, the main potential of SLM lies in the preparation of complex shaped products which machining might not be possible. Therefore, our paper is focused on the characterization of SLM Ti6Al4V alloy in the as-built condition with no surface treatment. The aim is to assess whether the application of HIP is reasonable in such case.

2. Materials and methods
This paper is focused on titanium alloy Ti6Al4V, a material widely used in medicine and aerospace industry [13]. For such applications, selective laser melting (SLM), as an additive manufacture technology, is useful as it provides geometrical freedom of manufactured products. We applied SLM to fabricate testing samples. As SLM does not yield completely defectless products, their mechanical behavior is influenced by present defects. Hot isostatic pressing (HIP) was thus applied to achieve ideal defect-free structure. The influence of HIP on the structure and mechanical response of testing samples was then assessed.

2.1. SLM
Selective laser melting of Ti6Al4V alloy was carried out on an M2 cusing machine by ConceptLaser. SLM setting was adjusted according to the manufacturer’s recommendation, table 1. Island scanning strategy, with island size of 5x5 mm², was applied. The whole manufacturing was carried out under a protective argon atmosphere (4.6 purity) to prevent oxidation. Ti ELI Grade 23 powder supplied by ConceptLaser was used. Its composition meets the ASTM F136-02a standard.

Round dog-bone samples with a gauge diameter of 5 mm, suitable for both tensile and fatigue tests, were manufactured. The longitudinal axis of the samples was oriented in the building direction Z. All samples were kept in the as-built state with no surface treatment.

| Table 1. SLM setting. |
|-----------------------|
| Laser power | Scanning speed | Hatching distance | Layer thickness |
| 200 W       | 800 mm/s       | 112 µm            | 30 µm          |

2.2. HIP
Hot isostatic pressing was done commercially at an argon pressure of 102 MPa at a temperature of 920°C for 2 hours.

In order to provide a comparison, a recommended standard heat treatment for Ti6Al4V alloy SLM products was done for one batch of the samples at 820°C for 1.5 hours. Because we registered a diffusion of oxygen during heat treatment under argon atmosphere (technical argon of 4.6 purity) [14], we carried out the heat treatment in vacuum (a vertical vacuum furnace Xerion XRETORT, 10⁻⁶ MPa).

For simplification, three studied states will be denoted further on as ‘no HT’, ‘sHT’ (standard HT) and ‘HIP’.

2.3. Metallography
Microstructure and porosity were studied on metallographic sections prepared by a standard metallographic way parallel to the building direction of SLM. The whole sections in un-etched condition were captured by optical light microscope (Olympus PME3) and processed by image analysis in ImageJ.
software to determine porosity. After etching in Kroll’s reagent, microstructure was observed by a scanning electron microscope Tescan Vega-3 LMU. The thickness of \( \alpha \)-lamellae in the \( \alpha+\beta \) microstructure was determined by means of image analysis.

2.4. Mechanical properties
Mechanical properties were studied under uniaxial tensile loading, under compression and during symmetric cyclic loading tension-compression with asymmetry ratio \( R = -1 \). Static loading was carried out on a universal testing machine LabTest 5.250SP1-VM at room temperature at a constant deformation rate of 0.001 s\(^{-1}\). High-cycle fatigue tests were measured at a frequency of 50 Hz according to ASTM E466-7 standard (universal servo-hydraulic testing machine Instron, force capacity 50 kN). The fatigue limit was determined after \( 10^7 \) cycles. To evaluate only the effect of microstructure on mechanical performance of the Ti6Al4V alloy (neglecting influence of porosity), Vicker’s hardness was measured on a semi-automatic hardness tester Future-Tech FM-700 with a load of 1 kg.

3. Results and discussion
The results section presents microstructural changes between the as-built state, after standard heat treatment and after the HIP of SLM Ti6Al4V alloy. The effect of HIP on porosity is shown. Static mechanical properties are compared for the initial state and both post-process treatments. In fatigue tests, only the effect of HIP is evaluated by testing sHT samples and HIPed samples.

3.1. Microstructure
Microstructures of the studied states of SLM Ti6Al4V alloy are presented in figure 1. In the as-built state, Ti6Al4V shows fully martensitic microstructure with acicular morphology of \( \alpha' \)-martensite (figure 1(a)). The diffusionless transformation results from high cooling rates during SLM, which can reach up to \( 10^6 \)°C/s [1]. When heat treatment is applied, with an annealing temperature higher than the temperature of martensitic decomposition (650–800°C [15]), martensitic phase transforms into a more stable \( \alpha+\beta \) microstructure (figures 1(b) and 1(c)). As the HIP treatment was characterized by a temperature of 100°C higher than the standard HT and 30 minutes longer dwell at such temperature, the thickness of \( \alpha \)-lamellae has grown to 2.1±0.8 µm compared to 0.8±0.3 µm in the sHT state.

![Figure 1. Microstructure of SLM Ti6Al4V alloy: (a) noHT, (b) sHT, (c) HIP.](image)

3.1.1. Porosity. The primary purpose of HIP is to eliminate porosity. The samples prepared by SLM showed porosity of 0.49±0.17%. The pores were of irregular shape, elongated in the scanning direction, sometimes containing particles of unmelted powder (figure 2). The origin of such pores is attributed to the lack of fusion due to an insufficient energy. For proper fusion between individual scan lines in one layer, it is necessary that melt pools formed by adjacent scan lines overlap. Similarly, proper fusion between two layers is ensured if the depth of melt pools is larger than the thickness of a deposited layer. Good melt pool overlapping is primarily determined by hatching distance which approximately tells
what is the overlapping between adjacent scan lines. However, the actual melt pool width is dictated by actual energy density (energy given to a volumetric unit of the powder). Energy density is a function of laser power, scanning speed, layer thickness, hatching distance, but the actual melt pool size is also dependent on powder properties, scanning pattern, preheating etc. [16]. HIP closed most of the pores, resulting in minimal porosity of 0.01±0.01%.

**Figure 2.** Longitudinal metallographic sections showing porosity in: (a) non-HIPed samples and (b) HIPed samples.

### 3.2. Mechanical properties

HIP affected mechanical properties of the SLM Ti6Al4V alloy by change in the microstructure as well as in porosity. Porosity lowers the load-bearing area and acts as stress concentrator, so negatively affects mechanical performance in both static and dynamic loading. Microstructural fineness of SLM as-built Ti6Al4V alloy and high internal stresses give it high strength, often higher than in conventional wrought condition [17,18].

#### 3.2.1. Static properties

Mechanical properties determined by static loading are summarized in table 2 (TYS – tensile yield strength, UTS – ultimate tensile strength, A – elongation, CYS – compressive yield strength, UCS – ultimate compressive strength, HV1 – Vicker’s hardness with 1 kg load). The highest yield strength, both in tension and compression, was reached in the as-built state with no HT. Very fine martensitic needles and high internal stresses associated with martensitic structure contribute to material strengthening. On the other hand, such material condition suffers from low elongation. Generally, heat treatment leads to the coarsening of the microstructure (transformation of martensite into lamellar α-phase and occurrence of interlamellar β-phase) and release of internal stresses, which reduces the material strength but promotes plasticity. In addition, the application of pressure during HIP closes the present porosity and promotes plasticity further. After sHT the elongation doubled, while it tripled after HIP. A minimal elongation required by ASTM F1472 standard for wrought Ti6Al4V alloy of 10% was surpassed only after HIP although standard HT brought the plasticity close to this limit.

**Table 2.** Mechanical properties of the studied states of SLM Ti6Al4V alloy.

|                | TYS (MPa) | UTS (MPa) | A (%)  | CYS (MPa) | UCS (MPa) | HV1 (-) |
|----------------|-----------|-----------|--------|-----------|-----------|---------|
| no HT          | 1000±21   | 1193±35   | 4.3±1.5| 1235±12   | 1763±30   | 389±9   |
| sHT            | 973±4     | 1023±4    | 9.0±2.0| 1079±6    | 1942±33   | 355±5   |
| HIP            | 946±12    | 1043±2    | 13.9±1.8| 1041±5    | 1766±30   | 330±6   |

* TYS – tensile yield strength, UTS – ultimate tensile strength, A – elongation, CYS – compressive yield strength, UCS – ultimate compressive strength, HV1 – Vicker’s hardness with 1 kg load

Despite a slight decrease in TYS, UTS, CYS and UCS due to the coarsening of microstructure, measured properties still satisfy the requirements by ASTM F1472 standard (min. TYS of 860 MPa and min UTS of 930 MPa). The difference of about 30 MPa between TYSs and CYSs of sHT and HIP state
are caused by larger thickness of α-lamellae after HIP (figure 1), although this difference is partially offset by the removal of porosity.

To assess only the influence of microstructure without the influence of porosity, we measured Vicker’s hardness with only 1 kg load. Increase in α-lamellae thickness from 0.8 to 2.1 µm after HIP led to a decrease in hardness by 25 HV. The transformation from martensitic needles (no HT) into α+β lamellar microstructure (sHT) brought a higher decrease by 34 HV as internal stresses were relieved. Generally, Vicker’s hardness can be used to roughly estimate material strength. The value of TYS should be approximately 3 HV [19]. If we look at the HIP samples and sHT samples, ΔHV of 25 should yield in a difference of ~ 75 MPa but the real difference is only 27 MPa because 0.49% porosity, which has the contradictory effect on strength, was present in non-HIPed samples.

![Fracture surfaces](image_url)

**Figure 3.** Fracture surfaces: (a,A) no HT, (b,B) sHT, (c,C) HIP.

Images of fracture surfaces (figure 3) clearly depict the above-described differences. With no porosity, the fracture surface of HIP samples (figure 3(c)) resembles a standard fracture surface of wrought material. The fracture surface is plane while in non-HIPed samples (figures 3(a) and 3(b)), it is rough because the crack propagated through present lack-of-fusion defects. It is nicely visible that these lack-of-fusion defects are arranged in a grid of parallel rows due to the incomplete overlap of melt pools in adjacent scan lines. In detail images (figure 3A-3C), a dimple-like morphology of fracture can be seen, with dimple depth increasing with increase in plasticity.

3.2.2. Fatigue. Although HIP is recommended to increase the fatigue limit of SLM Ti6Al4V alloy, our measurements on samples in as-built condition showed that the contribution of HIP is negligible. Non-HIPed samples in sHT state showed a fatigue limit of 212±10 MPa for the required lifetime of 10⁷ cycles. After HIP, the fatigue limit was 222±19 MPa. The explanation is clear from figure 4 showing fracture surfaces after the rupture. Samples loaded at a stress level of 300 MPa are compared. In both sample groups, initiation of fatigue crack occurred at the sample surface (as indicated by the arrows in figures 4(a) and 4(b) and detail images of initiation site in figures 4A and 4B). Machining the as-built surface is thus needed to achieve the desired effect of HIP.
Figure 4. Fracture surfaces after fatigue tests: (a,A) sHT, (b,B) HIP.

4. Conclusion
The key asset of SLM is the preparation of complex structures which cannot be achieved by conventional fabrication techniques. Such structures include porous structures (fully porous parts, surface porosity, internal porous structures) or hollow parts (enclosed, intricately shaped) that cannot be machined at all or with great difficulty. In such case, additional treatment by HIP is questionable. We have shown that without surface treatment the contribution of HIP to the fatigue limit of SLM Ti6Al4V alloy is negligible. The main contribution was an increase in elongation (13.9±1.8%) above the desired limit of 10% thanks to the closure of lack-of-fusion defects. Nevertheless, the coarsening of the α+β lamellar microstructure due to the higher temperature of HIP brought about a slight decrease in material strength and hardness.

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