

## MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HEAT TREATED SELECTIVE LASER MELTING MANUFACTURED Ti-6Al-4V

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### Abstract

*Investigation of microstructural evolution of pre and post heat treatment (HT) Selective Laser Melting (SLM) Ti-6Al-4V is presented. As received SLM samples composed of fine acicular  $\alpha'$  martensite within prior  $\beta$  grains were solution treated above the  $\beta$ -transus temperature, followed by a complementary HT within the  $\alpha+\beta$  range. Non-monotonic change of microhardness as a function of HT temperature was observed. After low HT temperatures, the microstructure was composed mainly of martensitic  $\alpha'$  phase, whereas, after high temperatures HT, bi-phasic microstructure with needle-like elongated  $\alpha$ -phase grains and a mixture of lamellar  $\alpha+\beta$  in between, was observed as the dominant microstructural component. The dependence of the properties and microstructure on heat treatment temperature will be discussed.*

**Key words:** SLM, Ti-6Al-4V, microstructure, thermal treatments, mechanical properties

### 1. INTRODUCTION

Ti-6Al-4V is classified as an  $\alpha+\beta$  alloy with 6 wt% Aluminium stabilizing the hcp  $\alpha$  phase and 4 %wt Vanadium stabilizing the bcc  $\beta$  phase. The microstructures are divided into several types, namely grain boundary  $\alpha$  ( $\alpha_{G.B.}$ ), primary  $\alpha$  (called bi-modal microstructure when the globular  $\alpha$  is surrounded by Widmanstätten plates), basketweave and martensite. Upon cooling from high temperatures (within or above the  $\alpha+\beta$  region), the  $\beta$  phase transforms into several different types of morphologies, which strongly depend on the applied cooling rate.

Diffusion controlled transformations take place at slow cooling rates ( $CR < 20$  C/s) (Ahmed and Rack 1998), resulting in bi-modal microstructure when cooled from the  $\alpha+\beta$  region. When cooled from the  $\beta$  region, initially,  $\alpha_{G.B.}$  is formed on the  $\beta$  grain boundaries, headed by nucleation and growth of parallel plates belonging to the same variant of the Burgers relationship inside the  $\beta$  grains, resulting in a Widmanstätten microstructure composed of several different orientation groups of parallel  $\alpha$  plates within the retained  $\beta$  phase. This microstructure is often referred to as lamellar, whereas each group of parallel plates is referred to as  $\alpha$ -colony. Slightly higher cooling rates favour nucleation over growth, resulting in a finer Widmanstätten structure composed of finer  $\alpha$ -colonies. Past research (Lütjering 1998) has shown that the colony size has a direct impact on mechanical properties.

High cooling rates ( $CR > 410$  C/s) induce diffusionless transformation ( $\beta \rightarrow \alpha'/\alpha''$ ) resulting in the formation of a martensitic phase which exists in two different structures: hcp distorted  $\alpha'$  acicular martensite and soft orthorhombic martensite -  $\alpha''$ . Acicular  $\alpha'$  is distorted due to Vanadium supersaturation, whereas  $\alpha''$  is poorer in Vanadium, hence less distorted. The stability of the  $\beta$  phase depends on the dissolution of Vanadium. At temperatures close to  $\beta$ -transus ( $T_\beta \approx 980^\circ\text{C}$ ), the  $\beta$  phase composed of 4 %wt Vanadium and upon quenching, acicular  $\alpha'$  is observed. Below the transus temperature, the  $\beta$  phase experiences a wide temperature-dependent compositional variation, as a lower temperature results in Vanadium enrichment of the  $\beta$  phase. Therefore, for low quenching temperatures, the  $\beta$  phase is stabilized and retained. This is the reason that, upon quenching from an intermediate temperature range of  $750^\circ\text{C}$ - $900^\circ\text{C}$ , orthorhombic  $\alpha''$  is expected to form in addition to some retained  $\beta$ . A mixture of both  $\alpha'$  and  $\alpha''$  is expected upon quenching from and above the intermediate temperature range, however for higher quenching temperatures, the  $\beta$  phase is poorer in Vanadium, thus the formation of distorted  $\alpha'$  is favoured. During HT, the martensite phase rejects the supersaturated Vanadium atoms which remain in excess of the solubility limit, leading to its decomposition to produce

thermodynamically stable  $\alpha+\beta$  phases. For further information on Titanium alloys, the reader is referred to Ref. (Banerjee and Mukhopadhyay 2010; Gerd Lütjering 2007; Matthew J. Donachie 2000).

Selective Laser Melting (SLM) is an Additive Manufacturing (AM) technology which allows to produce components layer by layer out of powdered metal according to a 3D-CAD volume model. Detailed description of the process is brought in Ref. (Chua 2010; Vandenbroucke and Kruth 2007). SLM offers several distinctive advantages over the traditional processing, such as its ability to produce complex geometrical components, high material use efficiency and Near-Net-Shape production. However, due to the high temperature gradient induced by the laser beam and the subsequent fast cooling, the resulting component is thermally stressed and composed of non-equilibrium phases. In order to improve the mechanical properties of SLM fabricated components and satisfy the industrial needs, post HTs are required.

Very few information is available on SLM post HT Ti-6Al-4V in the literature. The goal of this research is to investigate the As-Received (AR) SLM manufactured Ti-6Al-4V microstructure and its evolution induced by different HTs.

## 2. MATERIALS AND METHODS

The samples were manufactured by RAFAEL Israel using EOS M280 machine equipped with 200W, Yb-fibre laser. Standard manufacturer conditions, as described in Ref. ("EOS E-Manufacturing Solutions" 2015) were used to build the samples. Ti-6Al-4V 30 $\mu$ m powder with the composition given in Table 1, which corresponds to the ISO 5832-3, ASTM F1472 and ASTM B348 standards, manufactured by EOS, was used. All samples were stress relieved for 3 hours at 650°C in vacuum and furnace cooled (FC). These samples will be further addressed as the as-received (AR) samples.

Table 1. Chemical composition of the Ti-6Al-4V 30 $\mu$ m powder used to produce SLM samples

| Ti  | Al   | V    | O    | N     | H    | Fe   | C     | Y      |
|-----|------|------|------|-------|------|------|-------|--------|
| Bal | 6.19 | 3.98 | 0.12 | 0.018 | 0.02 | 0.18 | 0.004 | <0.001 |

Solution treatment of the AR samples was carried out for 1 hour at 1050°C in an atmospheric environment, followed by Water Quenching (WQ). These samples will be further addressed as Solution Treated and Water Quenched (STQ) samples. The HT was carried for two hours at the following temperatures: 950°C, 900°C, 850°C, 800°C, 750°C, 700°C and 650°C followed by FC with an approximate cooling rate of 0.02 C/s. These samples will be further addressed as Solution Treated, Water Quenched, Heat Treated and Furnace Cooled (STQHF) samples. The morphology was characterized by BSE mode in SEM (FEI Quanta 200 equipped with EDS), TEM (FEI Tecnai G<sup>2</sup> T20) and by optical microscopy (Reichert Polyvar). In order to reveal the microstructural features, the samples were etched with Kroll's reagent. The crystallography was examined by X-ray diffraction (Rigaku SmartLab) using Cu K $\alpha$  radiation in a parallel beam configuration. The microhardness was measured with Shimadzu microhardness tester with an applied force of 2kg for 15 sec. In order to obtain statistical information, each sample was measured at 7-15 different locations. Young Modulus was estimated by ultrasonic wave measurements (Epoch 4) and the Yield Stress was estimated by tensile test of a dog bone sample, with its axis of symmetry perpendicular to the building direction.

### 3. RESULTS AND DISCUSSION

#### 3.1. As received samples

The grain morphology of the Longitudinal Direction (LD) parallel to the SLM building direction and the Transverse Direction (TD) perpendicular to the building direction are shown in Figure 1.a and 1.b, respectively. The LD grain morphology was characterized by columnar grains elongated approximately parallel to the building direction. This grain morphology is typical of the SLM fabrication method (H. K. Rafi et al. 2015; Yan and Yu 2015). A high energy focused laser beam scans the deposited powder at a constant scan velocity. As the laser beam propagates, a steep temperature gradient is developed between the melted powder and its surroundings, leading to fast directional solidification as the previously deposited grains act as nucleation sites for epitaxial growth of the  $\beta$  phase. The rapid cooling from the  $\beta$  phase region induce non-equilibrium martensitic transformation  $\beta \rightarrow \alpha'$ , resulting in long orthogonally oriented martensitic plates with acicular morphology located at the elongated prior  $\beta$  grains. The TD grain morphology is characterized by prior equiaxed  $\beta$  grains (top view of the LD columnar elongated grains), which corresponds to the scan velocity of the laser beam composed of  $\alpha'$  martensite with similar morphological features acquired for LD.

A higher magnification of the martensite phase (Figure 1c) reveals twins associated with the  $\beta \rightarrow \alpha'$  martensitic transformations. Various authors (Banerjee and Mukhopadhyay 2010; Beladi, Chao, and Rohrer 2014; Matsumoto et al. 2013) reported the presence of dislocations and stacking faults within the  $\alpha'$  phase.

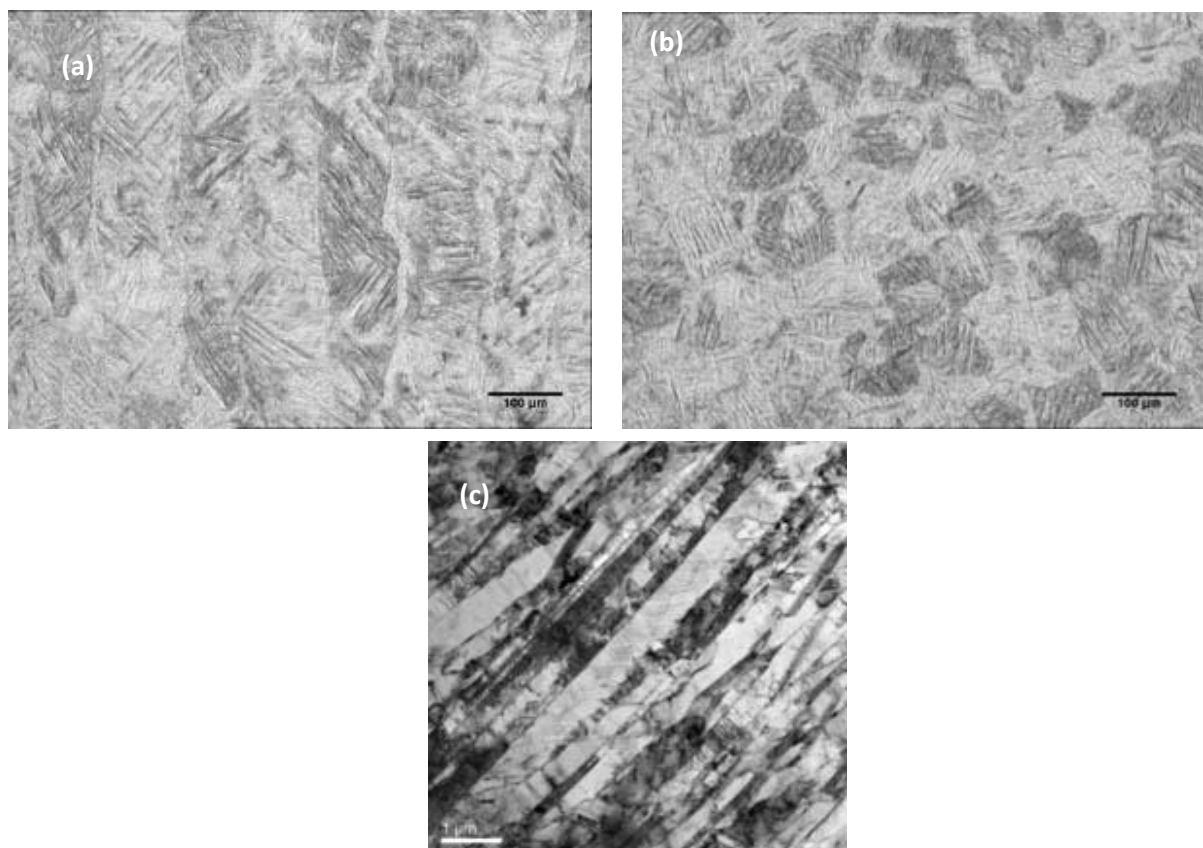


Figure 1: Microstructure of AR samples: (a) LD elongated grain morphology and (b) TD equiaxed grain morphology, in (c) TEM higher magnification of acicular  $\alpha'$ .

The XRD pattern demonstrates broad peaks of hcp reflections, whereas reflections from the  $\beta$ -bcc phase were not detected (Figure 2a). Martensitic  $\alpha'$  and  $\alpha$ -phase have a hexagonal lattice with similar cell parameters, therefore it is complicated to distinguish these two phases from each other (Jovanović et al. 2006). However, due to the fast cooling from the  $\beta$  region and the broadening of the peaks associated with microstrains developed during the martensitic transformations, it is reasonable to assume that the hcp phase is martensite.

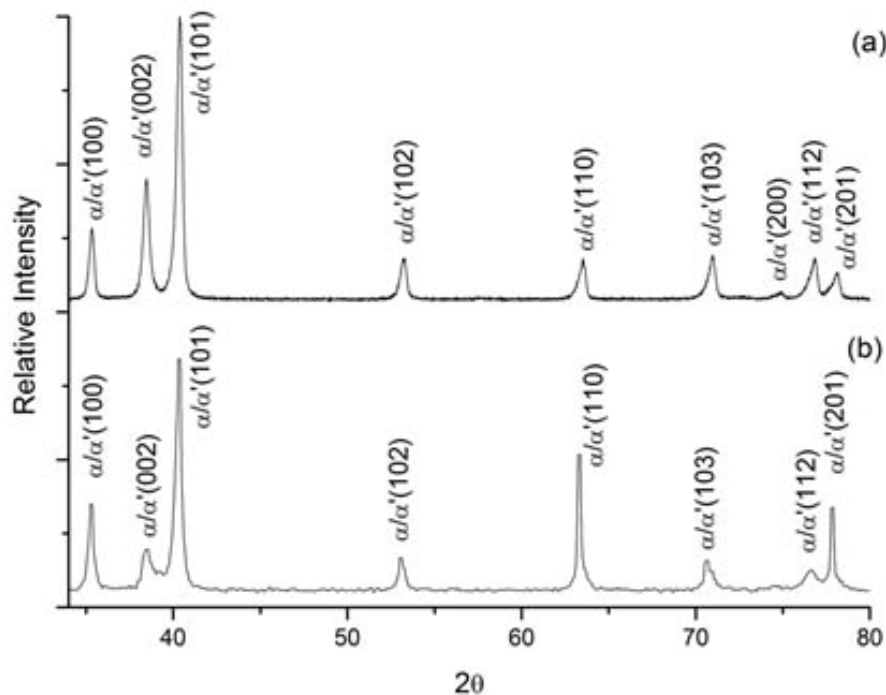


Figure 2: XRD patterns of (a) AR and (b) STQ samples

### 3.2. Solution treated samples

STQ-TD shows significant grain growth and discernible grain boundaries compared to the AR-TD microstructure (Figure 3a). Acicular martensite  $\alpha'$  was observed within the STQ grains. This result is in line with past research (Ahmed and Rack 1998), which has shown that solution treatment of 1 hour at 1050°C leads to complete decomposition of  $\alpha'$  within the  $\beta$  grains ( $\alpha' \rightarrow \beta$ ), which can grow in turn and transform into a new  $\alpha'$  martensitic phase upon WQ.

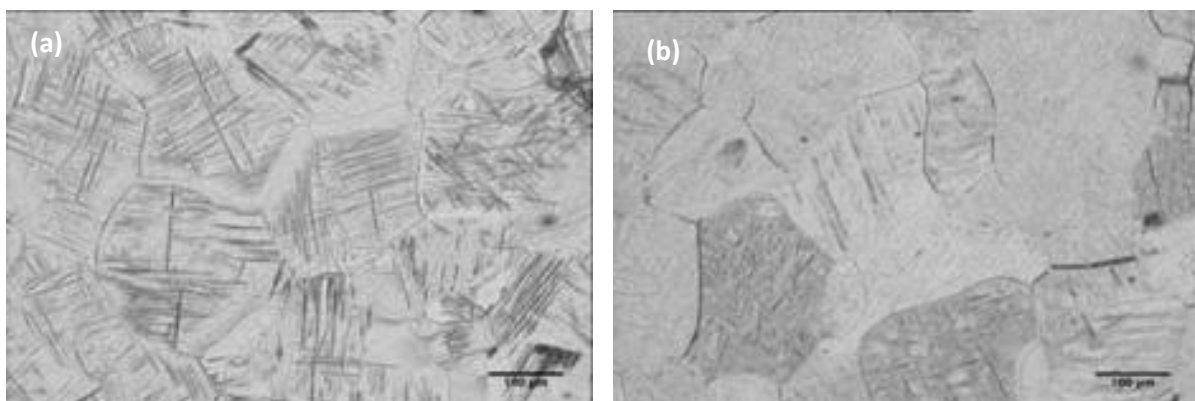


Figure 3: Microstructure of STQ: (a) aquiaxed morphology of TD and (b) LD.

The grain morphology of STQ-LD no longer contains elongated columnar grains and is composed mainly of equiaxial grains (Figure 3b). In order to investigate the LD grain morphology, two additional solution treatments were carried out. Samples held at 1050°C for 1 hour followed by FC or air cooling (AC) showed similar grain morphology with approximately the same typical dimensions. This leads to the conclusion that the grain morphology is independent of the cooling regime, hence the structural changes occur during the solution treatment above the  $\beta$ -transus temperature. This result contradicts the observations of (Vilaro, Colin, and Bartout 2011), claiming that the morphological change is due to a shearing mechanism which is related to martensitic transformations upon WQ from the  $\beta$  region.

One possible explanation for this observation can be correlated with the thermodynamically unstable  $\beta$  elongated columnar grains morphology, derived from the non-equilibrium SLM-fabrication. At 1050°C, the thermal energy is sufficient to induce recovery and nucleation processes accounting for the new equiaxial grain morphology.

At higher magnification, STQ samples exhibit a martensitic microstructure characterized by long orthogonally oriented plates with acicular morphology, as obtained for AR. Microstructural investigation to evaluate the needle size of STQ and AR is still in progress.

The STQ-XRD pattern (Figure 2b) exhibited broad Ti-hcp reflections. No presence of  $\beta$  phase was discernible. As mentioned in the previous section, due to the high cooling rates and relatively broad peaks, it is reasonable to assume that the hcp phase is martensitic  $\alpha'$ . Comparing the XRD pattern of AR to STQ, one can see the change in relative peak intensities, as can be vividly seen for {002} reflection. This observation is in line with the assumption that the nucleation process occurred during the isothermal holding at the  $\beta$  region. The newly formed grains have different crystallographic orientation, which results in different relative intensities acquired in STQ-XRD. For higher angles, a remarkable shift of STQ reflection towards lower angles is observed.

Microhardness measurements that were carried out for both AR and STQ samples are presented in Table 2. In their research (Vilaro, Colin, and Bartout 2011; Yadroitsev, Krakhmalev, and Yadroitsava 2014) obtained higher hardness values for AR compared to STQ, resulting from the finer plate size of  $\alpha'$  martensitic microstructure due to a high cooling rate which is typical of the SLM process. The higher microhardness of the STQ sample in the current study can be correlated with Oxygen dissolution during solutionizing in air. (Kumar et al. 2010) demonstrated a significant increase in microhardness of heat treated samples carried out in air due to Oxygen dissolution. This also explains the shift of hcp reflections in the STQ-XRD pattern to lower angles comparing to AR, due to the increase in the lattice parameters induced by oxygen dissolution within the Ti-hcp phase. Similar results were obtained by (Malinov et al. 2002).

No significant difference between the TD and LD samples was observed, which implies that within the statistical error, TD and LD have similar microhardness properties. Past research showed that SLM-fabricated LD has preferential tensile properties (H. Khalid Rafi, Starr, and Stucker 2013; Simonelli, Tse, and Tuck 2014), however the observed variation is rather small.

Table 2: Microhardness of AR and STQ samples

|               | AR [Hv 2kgs] |       | STQ [Hv 2kgs] |       |
|---------------|--------------|-------|---------------|-------|
|               | LD           | TD    | LD            | TD    |
| Microhardness | 370±2        | 376±5 | 400±5         | 386±9 |

### 3.3. Heat treated samples

STQHF LD and TD resulted in similar microstructural features, LD can be seen in Figure 4.

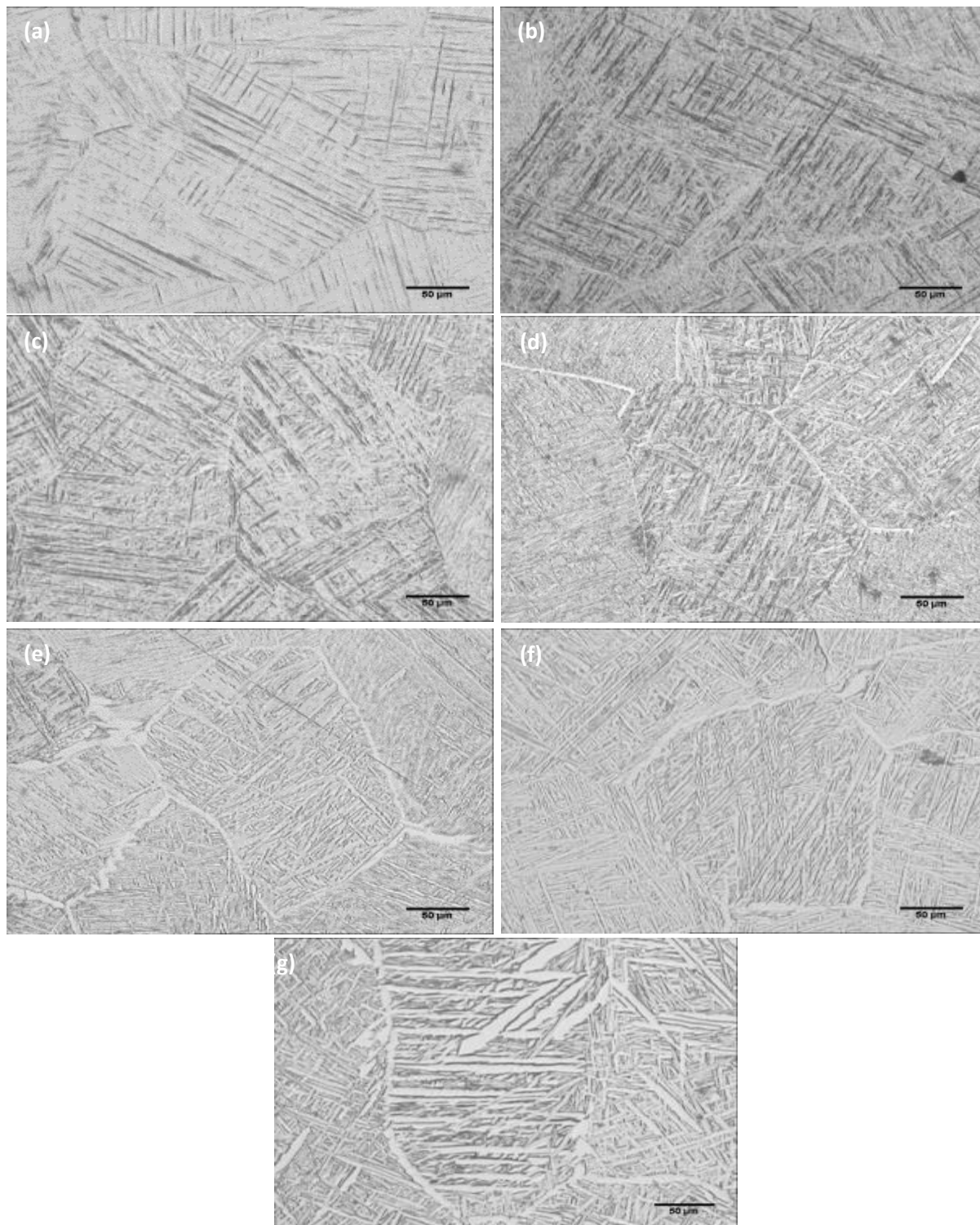


Figure 4: Microstructure of STQHF samples HT at: (a) 650°C (b) 700°C (c) 750°C (d) 800°C (e) 850°C (f) 900°C and (g) 950°C

Regardless of the HT temperature, similar size equiaxial primary  $\beta$  grains were observed. As expected, the size of STQHF grains corresponds with STQ since the sub-transus HT does not enable modification of the grain morphology.

Samples that were HT within the range of 650°C - 800°C (Figure 4a, Figure 4) exhibit typical martensitic microstructure comprised of long orthogonally oriented plates with acicular morphology. The BSE mode image of a sample heat treated at 650°C (Figure 5a) exhibits a uniform contrast within the sample, with distinct darker martensitic plates. The dark contrast is associated with different crystallographic orientations rather than variation in the chemical composition. Grain boundaries can be seen in samples that were heat treated at 750°C and above (Figure 4c to g), which is associated with precipitation of grain boundary- $\alpha$  phase ( $\alpha_{G,B}$ ) in prior  $\beta$  grain boundaries.

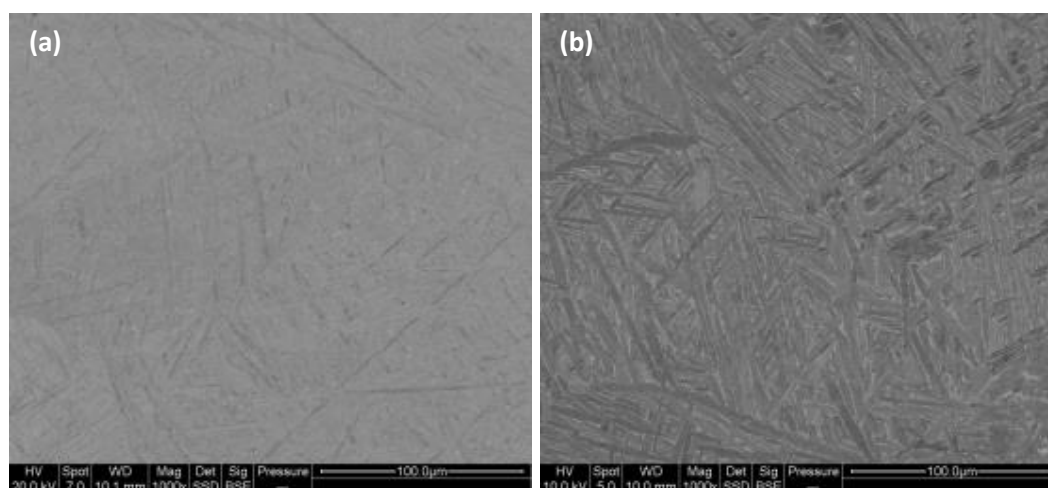


Figure 5: STQAF SEM-BSE mode of: (a) HT at 650°C - no compositional contrast can be seen (b) HT at 900°C – dark grey and bright grey contrast –  $\alpha$  phase, white contrast –  $\beta$  phase

In addition to martensite, elongated needle-like grains with typical  $\alpha$  phase compositions (acquired by EDS) can be seen in a sample heat treated at 800°C, which indicates partial decomposition of  $\alpha'$  and formation of a thermodynamically stable  $\alpha$  phase. A higher magnification revealed coarser plates with considerably less linear defects compared to AR and STQ samples (Figure 6). HRSEM-EDS and TEM-EDS composition investigation of samples heat treated below 800°C is currently in progress.

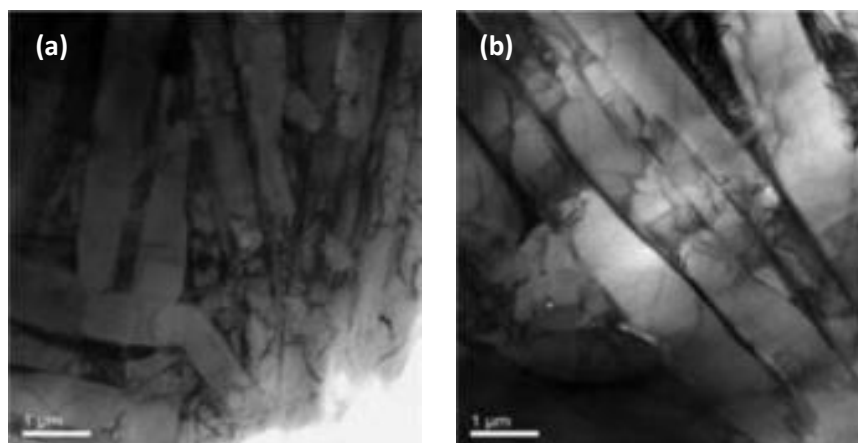


Figure 6: TEM images of samples heat treated at (a) 750°C and (b) 800°C

HT within the range of 800°C-850°C leads to high fracture martensite decomposition and subsequent formation of a biphasic microstructure (Figure 4e to g and higher magnification in Figure 7) composed of  $\alpha+\beta$  lamellar morphology located between long parallel needle-like  $\alpha$  grains, with very few colonies of untransformed martensitic plates. This result is in agreement with (Gil Mur, Rodríguez, and Planell 1996) observations. They showed that martensitic decomposition in Ti-6Al-4V is a time and temperature dependent process, corresponding with the Avrami equation. Full martensitic decomposition was achieved after HT at 800°C for 1 hour. The higher transformation temperature observed in the current study is expected due to oxygen dissolution in the samples mentioned in previous section. Oxygen is  $\alpha$  phase stabiliser (Gerd Lütjering 2007) which raises the  $\alpha \rightarrow \beta$  transformation temperature and subsequently the  $\alpha' \rightarrow \alpha+\beta$  transition. Increased HT temperatures (900°C-950°C) resulted in coarser biphasic microstructure. Similar microstructures were described by (Facchini 2010). BSE mode in a sample heat treated at 900°C (Figure 5b) demonstrated a vivid chemical composition difference between the darker, low Vanadium long parallel needle-like  $\alpha$  plates (as well as smaller  $\alpha$  plates within), and a Vanadium rich brighter  $\beta$  phase. EDS analysis at a range of 850°C-950°C (

Table 3) demonstrated typical compositions for the  $\alpha$  and  $\beta$  phases. No statistical difference between the needle-like  $\alpha$  and the lamellar  $\alpha$  could be revealed.

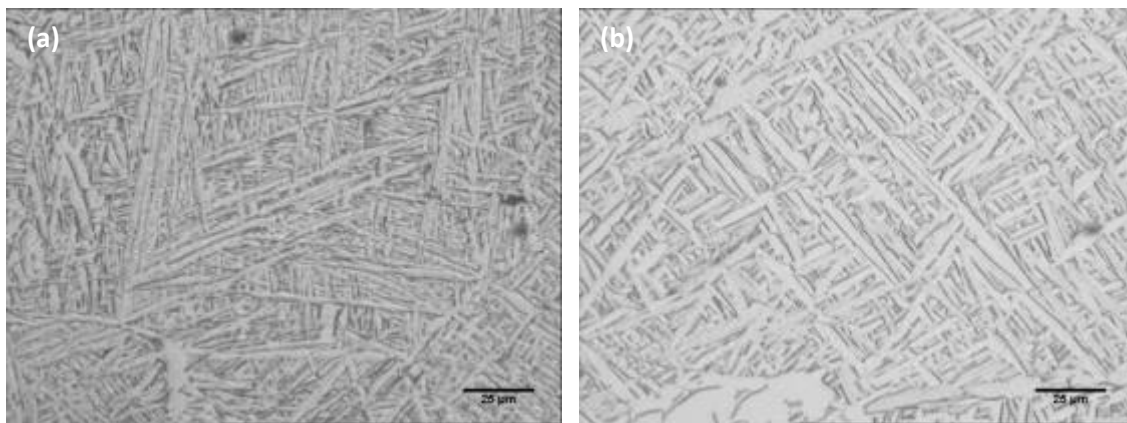


Figure 7: Microstructure of STQHF obtained at higher magnifications: images showing biphasic microstructure of samples heat treated at (a) 850°C and (b) 950°C.

Table 3: compositional EDS analyses of HT above 850°C

| Sample | $\alpha$ [%wt] |         |         | $\beta$ [%wt] |         |          |
|--------|----------------|---------|---------|---------------|---------|----------|
|        | Ti             | Al      | V       | Ti            | Al      | V        |
| 950°C  | 92±0.4         | 6.2±0.1 | 1.8±0.2 | 80.3±1.6      | 2.2±0.3 | 17.5±1.9 |
| 900°C  | 92.1±0.5       | 6±0.2   | 1.9±0.5 | 79.7±1.8      | 2.1±0.1 | 18.2±1.9 |
| 850°C  | 91.8±0.4       | 5.8±0.1 | 2.4±0.4 | 78.6±0.8      | 2.5±0.3 | 18.9±1.8 |

Microhardness measurements (Figure 8), characterized by a decrease of microhardness with the increasing HT temperatures (650°C-850°C) followed by a sudden increase at 850°C and a secondary decline with increasing temperatures (900°C to 950°C). A similar tendency was observed for TD microhardness measurements with no apparent statistical differences. The substantial microhardness drop observed at a HT range of 650°C-800°C is related to the microstructure coarsening, strain relief of martensite and its partial decomposition (mentioned in the previous paragraph).

The increase of microhardness obtained at 850°C resulted in high fracture martensite decomposition at this temperature, and subsequent formation of a bi-phasic microstructure composed of relatively fine lamellar  $\alpha+\beta$  morphology within elongated needle-like grains, as can be clearly seen in Figure 7a and b. The later decrease in microhardness (900°C-950°C) is explained by coarsening of the bi-phasic microstructure (See inserts in Figure 8), and reported by (Vrancken et al. 2012).

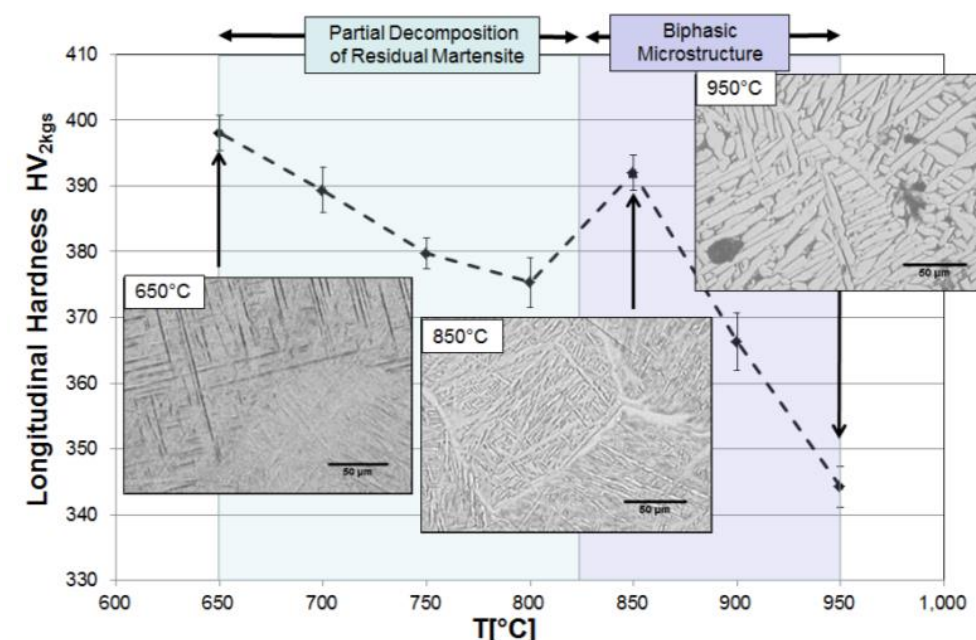


Figure 8: Microhardness measurements of STQHF samples, the attached OM presents a typical microstructure at different HT temperatures

Initial investigation to evaluate the tensile properties of STQHF samples heat treated at 800°C and 850°C was carried out and is presented in Table 4. The significantly higher yield stress obtained for a sample heat treated at 850°C is in agreement with microhardness observations, which indicates variation in the mechanical properties and further confirms the assumed phase transformation taking place within the range of 800°C-850°C. The elastic modulus of the two heat treated samples shows no significant difference.

Table 4: Tensile properties evaluation of STQHF

| Sample | Yield Stress [MPa] | Young Modulus [GPa] |
|--------|--------------------|---------------------|
| 800°C  | 850                | 112                 |
| 850°C  | 980                | 115                 |

## CONCLUSIONS

AR SLM manufactured Ti-6Al-4V samples exhibit distinct grain morphology difference. LD samples are characterized by fine long orthogonally oriented martensitic plates with acicular morphology -  $\alpha'$  within columnar prior  $\beta$  grains approximately parallel to the building direction, whereas TD is composed of similar to LD  $\alpha'$  microstructure within equiaxed prior  $\beta$  grains. Despite the morphological differences, both building directions suggest statistically similar microhardness values. Solution treatment above the  $\beta$ -transus modified the typical LD columnar prior  $\beta$  grain structure and resulted in a newly formed acicular  $\alpha'$  within coarser equiaxed prior  $\beta$  grains for both directions upon WQ.

Complementary sub-transus HT of the STQ samples were carried out, revealing microstructural changes and a non-monotonic variation of the microhardness values. HT at low temperatures (650°C-800°C) resulted in coarsening of the martensitic phase, its partial decomposition and subsequent decline of the microhardness values. HT at 850°C induced the transformation of metastable acicular martensite into thermodynamically stable biphasic microstructure composed of fine lamellar  $\alpha+\beta$  morphology, which is located between long parallel needle-like  $\alpha$  grains. The new fine biphasic microstructure gave rise to a sharp increase of microhardness. High temperature HT (900°C-950°C) led to coarsening of the biphasic microstructure and a decline of microhardness.

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