Tensile Properties of Vacuum Heat-treated Ti6Al4V Alloy Processed by Selective Laser Melting

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Abstract. This study presents an investigation on the tensile behaviour of heat-treated titanium alloy (Ti6Al4V) parts, which have been manufactured by Selective Laser Melting (SLM) additive manufacturing. The parts were heat treated at a temperature of 850ºC for 2 hours with and without Argon inert gas in a vacuum furnace. The tensile properties of the SLM samples with and without heat treatment have been compared to evaluate the mechanical performance of the 3D printed samples. The ultimate tensile strength (UTS) of as built Ti6Al4V part was found to be 1175 MPa. The UTS of heat-treated Ti6Al4V sample in vacuum without Argon gas was found to be 750 MPa, while the UTS of heat-treated sample in vacuum with Argon gas was recorded as 980 MPa. These values compare well with other published values of UTS for SLM built Ti6Al4V alloys with similar heat-treated conditions. The work also studied the micro-hardness of all the samples and compared the values with each other and found that the heat treated sample have lower average Vickers hardness values compared to as built samples.

1. Introduction

In recent years, Titanium (Ti) alloys have been the most used alloys in industries like aerospace and biomedical field because of their excellent properties like corrosion resistance, lightweight with high strengths. In particular, Ti6Al4V is currently identified as one of the strongest Titanium alloy used in commercial application. With the advent of additive manufacturing technology that offers the capability of manufacturing geometrically complex engineering components with high degree of efficiency, the production of complex, light-weight- high-strength components made from Ti6Al4V in a cost effective way is promising. Currently, a number of research utilizing additive manufacturing technology to produce components from Ti6Al4V have been performed [1-4]. However, due to the difference in additive manufacturing (AM) systems available within an organization, processing parameters employed and the geometry of the component produced, the likelihood of deriving the optimum processing parameters from the information available in literature is found to be limited. Therefore, more in-depth experimental study using the available AM technology in an organization to establish the optimum processing parameters including its post-processing route is required.

Selective Laser Melting (SLM) process is a laser based powder-bed type additive manufacturing process for 3D printing of metal parts using the metal powders using the digital model of the part [5]. The SLM uses the metal power with required size and properties, which helps SLM process to deliver the desired
properties of metals. Selective laser melting process uses a laser beam to scan and melt the fine metal powdered particles spread on a platform with powder undergoing rapid melting and solidification. The process scans the cross-section of each layer of powder according to digital model on the surface of the bed. The process is repeated until the final part is completed.

Several researchers have investigated the mechanical properties of Ti6Al4V parts manufactured by Selective Laser Melting (SLM) additive manufacturing technology. They have studied the tensile, fatigue, porosity and micro hardness of Ti6Al4V with or without the heat treatment of the SLM built parts. The properties of the SLM built parts depends not only upon careful selection of various SLM parameters but also on a variety of post-build heat treatments. Qiu et al [6] investigated the effect of laser power, laser scanning and orientation on the tensile properties of Ti6Al4V samples produced from Selective laser melting (SLM). The as-built samples exhibited high tensile strengths but poor ductility with elongation generally less than 10%. The hot isostatic pressing (HIP) reduced the tensile properties but increased elongation compared to as-built samples. Facchini et al [7] studied the changes in the mechanical properties particularly ductility along with the microstructure of Ti6Al4V alloy produced by selective laser melting (SLM). They found micro cracks in the samples due to the effect of incomplete homologous wetting and residual stresses produced by the large solidification under cooling of the melt pool. For as-built SLM parts, the ultimate tensile strength was found to be 1095MPa, the yield stress was 990 MPa, and breaking elongation was 8.1%.

Thone et al [8] have done tensile and fatigue properties of selective laser melting (SLM) produced Titanium alloy (Ti6Al4V) made on SLM250HL machine from SLM Solutions. The samples were heat treated for 2 hours at temperatures between 750°C to 850°C in Argon atmosphere followed by furnace cooling and some samples between 950 C and 1050 C in vacuum followed by furnace cooling. The tensile strength went on decreasing as the heat-treated temperature increased. Huang et al [9] investigated tensile properties of titanium alloys fabricated on Renishaw AM250 SLM machine for different heat treatment conditions, which included different temperatures and duration in Argon followed by water quenching or air cooling or furnace cooling. They found that the SLM sample with heat treatment at 800C for 2 hours followed by air cooling had the highest breaking elongation of 17% compared to 5.37% for as-built sample with no heat treatment. Vrancken et al [10] have also studied the optimization of the mechanical properties through heat treatments of the titanium alloy (Ti6Al4V) produced on an in-house developed LM-Q SLM machine of the PMA Division of the Department of Mechanical Engineering, KU Leuven. In that study, the effects of several heat treatments in a vacuum oven with different cooling regimes on the microstructure and mechanical properties of Ti6Al4V were investigated. Results show that post heat treatment at 850°C for two hours, followed by furnace cooling increased the ductility of SLM parts to 12.84%, compared to 7.36 % for as-built parts.

Published literature indicates that only a few studies have been made to understand the effect of heat treatment on tensile properties of SLM processed titanium alloy. As more and more commercial SLM machines are being available offering a variety of process parameters, it is important to undertake wider studies on examining the mechanical properties of different alloys processed by selective laser melting.

In this study, the effect of heat treatment in vacuum and Argon atmosphere on tensile properties of Ti6Al4V parts processed by ProX200 Selective Laser Melting machine from 3D Systems is presented. A comparison of obtained results is made with the results of other similar studies of the same alloy fabricated on other SLM machines.
2. Experimental Work

2.1. Selective Laser Melting and Heat Treatment

In this study, 6 tensile test specimens with dimension complied with ASTM E-8/E8M-09 standard were manufactured from Ti6Al4V powder using ProX200 Selective Laser Melting system built horizontally flat in X-Y plane. The main processing parameters implemented are listed in Table 1.

| Parameter                  | Value       |
|----------------------------|-------------|
| Laser power (W)            | 215         |
| Scan speed (mm/s)          | 1800        |
| Hatch spacing (µm)         | 85          |
| Defocus distance (mm)      | 2.5         |
| Layer thickness (µm)       | 30          |
| Scan pattern type          | Parallel vectors at 45° angle |
| Scanning angle             | -45/45      |
| Gas used                   | Argon       |

Table 1. Selective laser melting process parameters in ProX200 Selective Laser Melting machine

After the manufacturing using SLM system, before removing the samples and their supports (anchors) from the built plate, the entire plate with all samples was stress relieved by heating in an open air oven for 2 hours at 650°C and then cooled down to avoid warping of samples due to residual stresses generated during SLM processing. The tensile test specimens then were cut off from the plate (Fig. 1).

![Figure 1: As built Ti6Al4V samples built on ProX200 Selective Laser Melting](image)

Following the manufacturing of tensile test specimens, 4 specimens were heat-treated in Nabertherm RHTH 120-300 vacuum furnace. The sequence of heat treatment was heating the specimens to 850°C in 4 hours followed by holding temperature at 850°C for 2 hours and finalized by cooling inside the furnace for 6 hours. During the heat treatment stage of the four specimens, two specimens were covered by Argon gas environment and the other two specimens were not covered by Argon gas environment. Table 2 explains the conditions of the three sets of tensile specimens (A, B and C) used in this study.

2.2 Mechanical testing and Microstructure analysis

Tensile tests were performed on the MTS criterion model 43, shown in Figure 2, with applied deformation rate of 1.0 mm/min. As-built and as-heat-treated tensile test specimen were longitudinally cross-section cut and mounted for grinding and polishing prior to hardness and metallography analysis. Grinding of specimens started using 320 grid, gradually increasing the grid size until and finalized with 1200 grid sand paper. Grinding process was followed by chemical-mechanical polishing with a mixture
of colloidal silica (OP-S) and hydrogen peroxide (30%). In order to reveal the microstructures, the surface of mounted samples was immersed in hydrogen peroxide solution at 60ºC for 25 seconds. Vickers micro-hardness testing was carried out at 12 locations across the centre of the longitudinal cross-section of tensile test specimen, under a load of 300g.

Figure 2 MTS Criterion Model 43 machine for tensile testing experiments

Table 2. Tensile test specimen conditions

| Specimen code | Conditions |
|---------------|------------|
| A             | As built on SLM, no heat treatment applied. |
| B             | As built on SLM, heat-treated in a vacuum furnace at 850ºC for 2 hours without Argon gas and followed by furnace cooling to room temperature. |
| C             | As built on SLM, heat-treated in a vacuum furnace at 850ºC for 2 hours with Argon gas and followed by furnace cooling to room temperature. |

3. Result and discussion

Figure 3 shows the variation of tensile properties and microhardness of the three sets of SLM made specimens under conditions described in Table 2. From figure 3, it is noted that the as-built specimen exhibits the highest strength (1,175 MPa) and highest hardness compared to the strength and hardness of heat-treated specimens. This result is predominantly contributed by acicular martensitic, α’ phase (bright colour structure) (Figure 4) formed as the result of rapid cooling rate during selective laser melting process [11, 12]. However, the as-built specimen exhibits trans-granular fracture with network of dimples (Figure 5), indicating ductile rupture, consistent with high elongation of 16.6% (Figure 4).
Heat treatment at 850°C, which is below β transus transformation [10], for 2 hours, allows the decomposition of α’ phase to α+β in heat-treated specimens to occur (Figure 6). In contrast to heat treatment at a temperature above β transus, when as-built specimen exposed to 850°C temperature, only a fraction of acicular α’ phase was decomposed that results in a mixture of α’+α+β phase to occur as the final microstructure. This mixture of α+β microstructure contributes to the lower UTS [13] and hardness and results in slightly lower ductility compared to the ones in as-built specimens (Figure 3). In Figure 7, it is noticeable that heat-treatment without Argon gas environment exhibits more trans-granular cracking with less dimple network, indicating lower ductility of 16.1% (Figure 3) compared to as-built specimen.

Figure 3. Tensile properties and micro-hardness of as-built and heat-treated specimens (A, B and C).

Figure 4. Microstructure of as-built specimen

Figure 5. Fracture surface of as-built specimen
Heat treatment at 850°C with Argon gas environment retarded the growth of α and β phases, thus producing smaller grain size (Figure 8) compared to heat treatment without Argon gas environment. As a result, an improvement in UTS is clearly observed (Figure 3). In addition, fracture surface of specimen heat-treated with Argon gas environment exhibited pockets of fine dimples, indicating an increase in ductility network that is consistent with an increase in elongation to 17.6% (Figure 3).

Table 3 shows a comparison of some tensile properties of Ti6Al4V alloys processed on different SLM machines but with almost similar heat treatment conditions. In general, the ultimate tensile strength of as-built specimens is higher but heat treatment tends to reduce the UTS values. In most cases, the heat treatment tends to increase the elongation at break, though this also depends upon various process parameter settings and build orientation.
Table 3. Comparison of Tensile properties of SLM processed Ti6Al4V alloys

| Heat treatment condition of Ti6Al4V | SLM Machine   | Young’s Modulus (GPa) | Ultimate Tensile Strength (MPa) | Elongation at Break % | Reference  |
|-------------------------------------|---------------|----------------------|-------------------------------|-----------------------|------------|
| As built                            | SLM250HL      | NA                   | 1080                          | 1.6                   | [8]        |
| As built                            | Renishaw AM250| NA                   | 1191                          | 5.37                  | [9]        |
| As built                            | LM-Q SLM      | 109                  | 1267                          | 7.28                  | [10]       |
| As built                            | ProX200       | 111.9                | 1175                          | 16.6                  | This study |
| Heat treated at 850°C for 2 hours with Argon and furnace cooled | SLM250HL      | NA                   | 1009                          | 5.2                   | [8]        |
| Heat treated at 800°C for 2 hours with Argon and air cooled | Renishaw AM250 | NA                   | 1073                          | 17.05                 | [9]        |
| Heat treated at 850°C for 2 hours in vacuum and furnace cooled | LM-Q SLM      | 114.7                | 1004                          | 12.84                 | [10]       |
| Heat treated at 850°C for 2 hours in vacuum and furnace cooled | ProX200       | 102.4                | 750                           | 16.1                  | This study |
| Heat treated at 850°C for 2 hours with Argon and furnace cooled | ProX200       | 103.26               | 980                           | 17.6                  | This study |

4. Conclusions

The aim of this investigation was to study the tensile properties of the Ti6Al4V alloy processed by the SLM process when subjected to heat treatment at 850°C in vacuum with or without the Argon inert gas. Specimen heat-treated at 850°C with Argon environment generated the best combination of strength and ductility compared to as-built specimen and heat-treated specimen without Argon gas due to the formation of finer grain of α+β microstructure. In terms of the tensile properties, the as built sample A was found to have highest ultimate tensile strength (UTS) of around 1175 MPa. It was noted that the heat treatment in vacuum reduces the tensile strength as observed by other published literature. It is also shown that the heat treated sample B has the ability to withstand more energy up to the level where the fracture starts. The heat-treated sample C with Argon showed the highest elongation value when compared to the other two samples. The higher UTS and lower ductility observed in the as-built specimen is contributed by acicular martensitic (α) phase while lower UTS but the higher ductility in heat-treated specimens is influenced by the formation of β-phase as a result of exposure to 850°C, a temperature below β transus transformation.

5. References

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