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Tensile properties and microstructural characterization of additive manufactured, investment cast and wrought Ti6Al4V alloy

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Abstract. This paper presents an evaluation of the tensile properties and microstructural characterization of Ti6Al4V alloy manufactured with three different processing routes; traditional wrought processing, investment cast and Laser Engineered Net Shaping (LENS). Tensile specimens were machined from each process and tensile tested at room temperature. Fractured specimens were characterized using light optical microscopy, stereo microscopy, microhardness and Scanning Electron Microscopy (SEM) to investigate the microstructural morphology and the structural hardness variation. The investment cast Ti6Al4V alloy microstructure revealed large equiaxed grains containing various orientated lamellar colonies. The additive manufactured microstructure revealed long columnar grains with Widmanstätten α' martensite laths and retained β grain boundaries. While the wrought Ti6Al4V microstructure was observed as smaller equiaxed grains with large colonies of fine lamellar and transformed β . Additive manufactured specimens had higher yield strength, ultimate tensile strength and hardness compared to the investment cast and wrought manufactured specimens.

1. Introduction

Titanium alloys have found many uses in the industries of aerospace, medical implants, medical equipment and chemical plants, this is because of its high strength to weight ratio and corrosion-resistant properties [1]. The Ti6Al4V alloy is commonly used in these industries for complex components, currently often manufactured with investment casting (IC) or thermomechanical processing routes [2]. The IC process is the most developed near-net-shape manufacturing process [3] and the main route for complex titanium products. Thermomechanical processing such as forging, rolling or compression carried out at elevated temperatures can improve the ingot's structure from brittle and porous with a coarse microstructure into a wrought structure with a finer microstructure giving better mechanical properties [4]. Laser Engineered Net Shaping (LENS), an additive manufacturing (AM) method has become a strong competitor in the production of titanium products due to its ability in creating near-net-shape products in less time. The process is cost-



effective in producing near-net-shape products, it has excellent material utilisation and gives an appealing fine microstructure [5], [6]. Each of these manufacturing methods has its own advantages and shortcomings. The challenge is to determine which manufacturing process is best suited for a specific component based on the mechanical properties required. The mechanical properties are dependent on the microstructures, while the microstructures are influenced by the manufacturing process. The Ti6Al4V alloy's microstructure is particularly sensitive to the manufacturing process and thermal history [5]. Thus, it is vital to study the effect of manufacturing processes on the microstructure of the alloy to understand and predict the mechanical properties of the alloy.

Although the microstructures and mechanical properties of IC, AM and wrought manufacturing have been studied individually, little attention has been given to an analytical comparison study between these manufacturing processes with respect to a single material, Ti6Al4V. Therefore, this paper is aimed at comparing the microstructures and tensile properties of wrought, IC and AM Ti6Al4V products.

2. Experimental

Ti6Al4V wrought bars were bought from Boati in China (Figure 1 a), the chemical composition for the as-received bar is given in Table 1. Wrought rods were wire cut from the as-received Ti6Al4V wrought bar (Figure 1 a).

Table 1. Chemical composition of the as-received wrought Ti6Al4V bar (wt. %)

Al	V	Fe	Y	C	H	N	O	Other	Ti
6.75	4.5	0.30	≤0.005	≤0.08	≤0.0125	≤0.05	≤0.2	≤0.4	Bal

The investment cast (IC) started with a wax tree pattern for eight rods of 212 mm length and 15 mm diameter (Figure 1 b) which was dipped into two alternating ceramic slurries, colloidal ZrO₂ and stuccoing with ZrO₂ stucco. The mould (Figure 1 c) was dried for 24 hours at room temperature. The wax burned-out was performed at 200°C and 8 bars pressure using a Leeds and Bradford Boiler (LBBC) Steam Boilerclave® for 15 minutes and then fired for two hours at 800°C. The as-received Ti6Al4V wrought bars (Figure 1 a) were melted using an induction melting furnace, the molten alloy was poured into the ceramic shell and left to solidify. The shell was broken off revealing the runners/sprue and Ti6Al4V investment cast rods (Figure 1 d). The IC rods were cut-off from the runners and sprue (Figure 1 e). The chemical composition of the Ti6Al4V IC rods is given in Table 2. Between

Table 2. Chemical composition of Ti6Al4V investment casting (wt. %)

Al	V	Fe	Y	C	Cr	Mo	Ni	S	Sn	Cu	Zr	Ti
6.76	4.51	0.12	ppm≤7	0.018	0.12	0.009	0.007	≤0.005	ppm≤50	0.021	ppm≤100	Bal

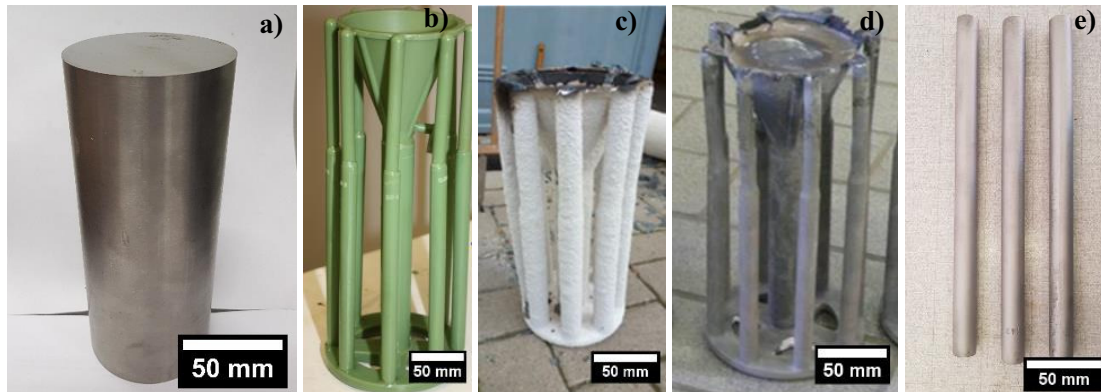


Figure 1. a) As received Ti6Al4V wrought bar, b) the wax tree pattern, c) ceramic mould, d) runners and Ti6Al4V investment cast rods, and e) Ti6Al4V investment cast rods.

Two as-received wrought Ti6Al4V alloy bars were supplied for atomisation to TLS (Technik GmbH & Co. Spezialpulver) an international supplier of titanium powder in Germany. Electrode Inert Gas Atomisation (EIGA) was used to produce spherical powder for additive manufacturing. EIGA is done by induction melting of a rotating electrode and atomised by inert gas [7]. The spherical atomized Ti6Al4V powder (Figure 2) and Energy Dispersive X-ray Spectrometer (EDS) analysis of some single particles of the atomized Ti6Al4V powder is given in Table 3.

The powder produced is spherical with a large range of particle sizes.

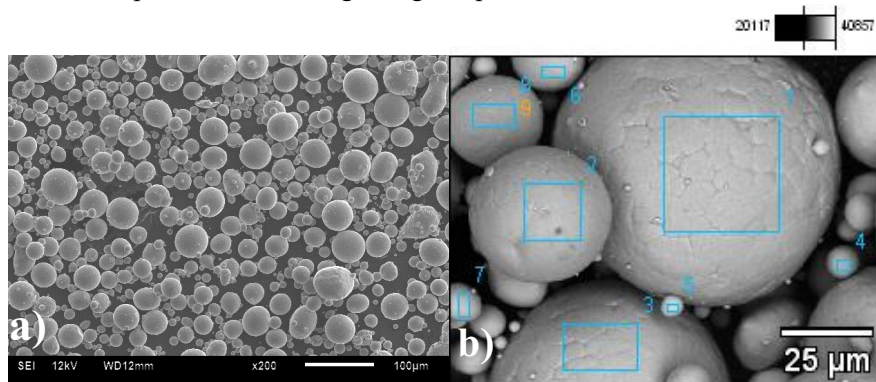


Figure 2. a) Spherical Ti6Al4V atomized powder, and b) Electron microscopy EDS analysis on some single particles

Table 3. EDS analysis of some single particles of atomized Ti4Al4V powder

Element	Al-K	Ti-K	V-K
Atomized Ti6Al4V _pt1	5.5	90.1	4.4
Atomized Ti6Al4V _pt2	5.5	90.1	4.3
Atomized Ti6Al4V _pt3	4.7	89.6	5.6
Atomized Ti6Al4V _pt4	4.3	91.5	4.2
Atomized Ti6Al4V _pt5	1.3	93.5	5.1
Atomized Ti6Al4V _pt6	3.5	92.6	4.0
Atomized Ti6Al4V _pt7	4.4	91.0	4.6
Atomized Ti6Al4V _pt8	6.1	89.5	4.4
Atomized Ti6Al4V _pt9	5.8	90.2	3.8

The atomised powder was used with the Optomec LENSTM 850-R system to laser print Ti6Al4V rods. The rods were built in the Y direction as seen in Figure 3 b. The Optomec LENSTM used a 1 kW IPG high energy fibre laser and three co-axial powder nozzles which are set at 8 ± 2 mm from the substrate (stand-off distance). The substrate was a 5 mm thick Grade 5 titanium plate which was sandblasted and cleaned with acetone to remove any surface contamination. The chamber was filled with argon gas to reduce oxidation, this argon gas also played the role of quenching medium as it was fed at room temperature. An energy density range of 180 J/mm^3 to 315 J/mm^3 was used to 3D print the Ti6Al4V rods.

The as-received Ti6Al4V wrought rods were heat-treated at 1050°C for 30 minutes then furnace cooled to generate colony lamellar structure, however, the IC Ti6Al4V rods were cut and hot isostatically pressed (HIPed) to reduce the porosity. While the AM rods were cut from the substrate in as 3D printed conditions. The tensile specimens were machined from IC and HIPed, heat-treated wrought and as 3D printed AM rods according to ASTM E 8M-04 standards (Figure 3 b). Tensile tests were carried out using a servo-hydraulic fluid-controlled machine (Instron 1342). Specimens were pulled at 0.25 mm/min at room temperature (19.80°C). The initial length, area, continuous load and extension values were captured, and the stress-strain curves drawn.

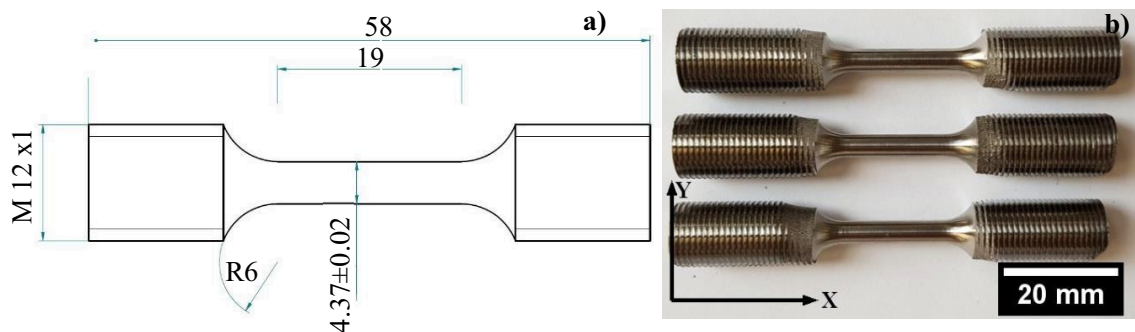


Figure 3. a) Schematic of the tensile specimen, and b) machined tensile specimens

Wrought, IC and AM samples were cut, mount and ground then polished and etched using Kroll's reagent to reveal the microstructure. The microstructure analysis was performed using the Leica DMI5000M Optical Microscope (OM) equipped with an image analysis software and Scanning Electron Microscope (SEM) equipped with energy-dispersive X-ray spectroscopy (EDX).

Microstructures were quantified using an image processing program (ImageJ). The grain sizes were measured using the line intercept method. The volume fraction of alpha-phase (α -phase) and beta-phase (β -phase) were measured using the segmentation method.

An automatic Vickers micro-hardness tester FM-700 was used to measure the change in hardness to investigate the structural homogeneity of the different manufacturing processes. Indentations were made on wrought, IC and AM specimens using a load of 500 g for a dwell time of 15 seconds.

Tensile fractures were studied using the Leica MZ16 Stereo-microscope and Scanning Electron Microscopy (SEM) to reveal the fracture mode.

3. Results

The microstructure of wrought, investment cast (IC) and additively manufactured (AM) Ti6Al4V specimens is shown in Figure 4 a, b, and c, respectively. The wrought Ti6Al4V microstructure shows small equiaxed grains, with blocky plate-like α/β -phase inside the grains (Figure 4 a). The IC grains have α -phase grain boundaries (prior β), the grains are equiaxed with diameters up to 1 mm (Figure 4 b). The grain boundaries, colonies and layer-bands of the AM microstructure are revealed in Figure 4 c.

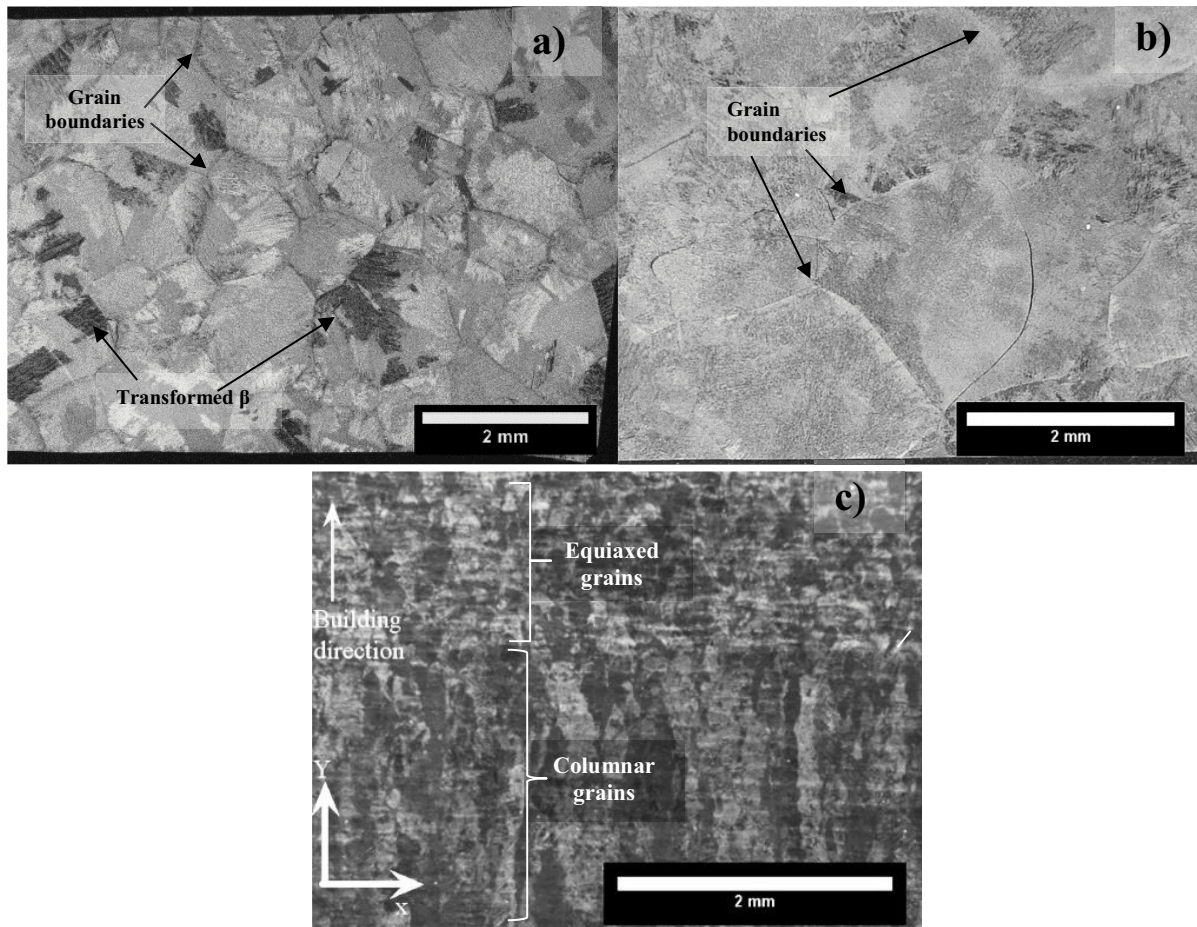


Figure 4. Grain boundaries, equiaxed and columnar grains respectively observed in a) wrought b) investment cast, and c) additively manufactured Ti6Al4V specimens.

The average grain size and the volume fraction of α and β -phase in wrought, investment cast and additively manufactured Ti6Al4V specimen are given in Table 4.

Table 4. Average grain size and volume fraction of α and β -phase

Manufacturing process	Average Grain size ($\mu\text{m}/\text{grain}$)	Average %	
		α	β
Wrought	551.1	65.0585	34.9415
Investment Cast	1109.2	64.273	35.727
Additively manufactured	146.7	65.214	34.786

The microstructural features of the wrought, investment cast and additively manufactured Ti6Al4V are revealed in Figure 5 a, b and c, respectively. Fine α/β lamellae, blocky plate-like and α/β colony structure are observed in Figure 5 a. Coarse α/β -lamellae colonies in various directions with blocky plate-like α (transformed β) are observed in IC Ti6Al4V Figure 5 (b). Columnar grains, Widmanstätten α' martensite laths with retained β - grain boundaries are revealed in AM Ti6Al4V.

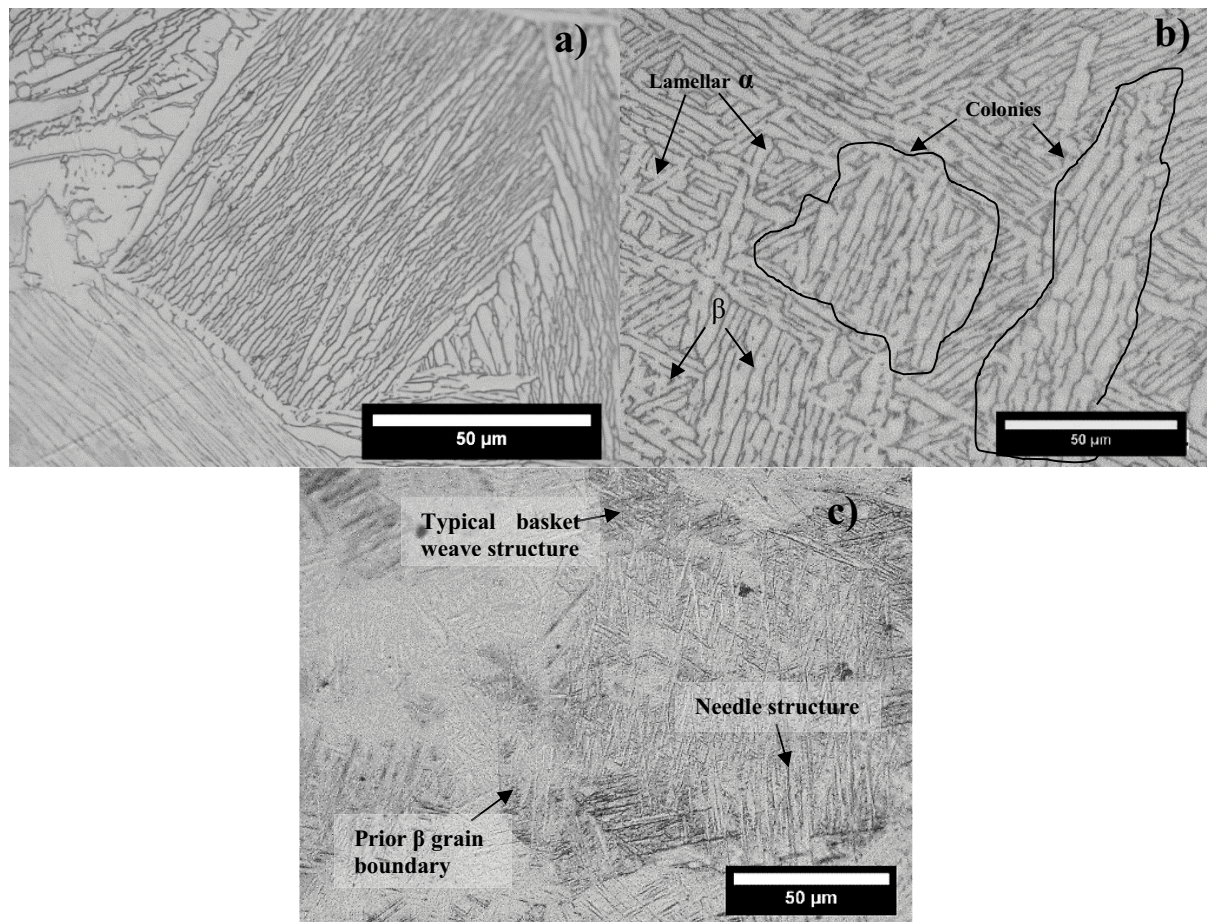


Figure 5. Optical microscope images of a) Wrought b) Investment cast and c) Additive manufactured

The Vickers micro-hardness profile of the wrought, IC and AM Ti6Al4V are given in Figure 6. The results clearly illustrate that the hardness varies for different manufacturing processes. The AM with a partial martensitic microstructure gave the highest average hardness value (408.8 HV0.5). The wrought and IC Ti6Al4V alloy specimens with colony lamellar structure gave similar average hardness of 341.8 HV0.5 and 320.3 HV0.5, respectively.

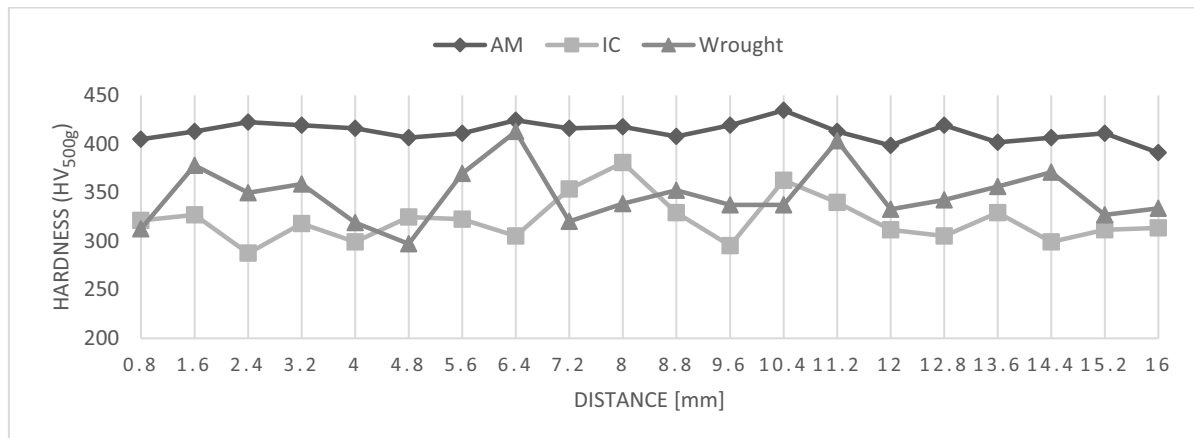


Figure 6. Hardness profile of AM, IC, and wrought Ti6Al4V structures

The stress-strain curves for tensile tests of wrought, IC and AM Ti6Al4V are given in Figure 7. The AM Ti6Al4V specimen had superior tensile properties with leading values in ultimate tensile strength (UTS) and yield strength (YS). However, the tensile elongation for AM is low (less than 10 %) as compared to the wrought and IC Ti6Al4V tensile specimens (higher than 10 %). The slopes are very similar, with the AM slope observed to be somewhat lesser steep compared to wrought and IC. The gradual slope of the AM specimen indicates a low modulus of elasticity. The wrought and IC specimens had the steepest slopes and relatively high % El (Percentage elongation), which is in agreement with Figure 8 a and b showing a larger necking for wrought and IC specimens and not any for AM specimens (Figure 8 c).

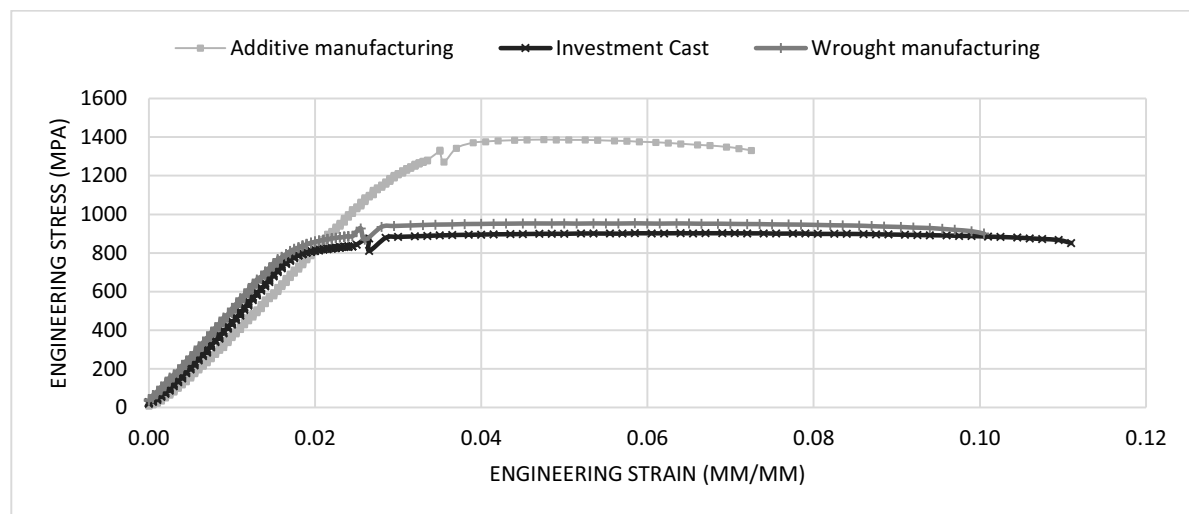


Figure 7. Stress-Strain plots of the IC, AM and wrought Ti6Al4V specimens

The tensile fractures of wrought, IC and AM Ti6Al4V tested specimens are shown in Figure 8. The wrought and IC fractures have somewhat necking (Figure 8 a, b), while the AM fracture has no necking (Figure 6 c).

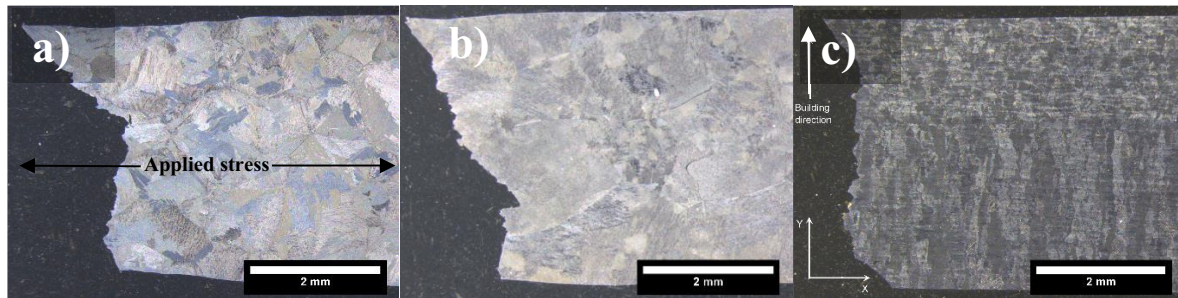


Figure 8. Stereo-microscope images of cross-sectioned tensile test fractures fabricated by a) wrought b) IC and c) AM processes

Fracture cross-sections of the wrought, IC and AM Ti6Al4V are given in Figure 9. Figure 9 a and b show kinking of α/β lamellae, the lamellar lines tend to deflect towards a fractured edge, in the ductile IC and wrought structure especially in the necking region of the tensile specimens. Figure 9 c illustrated a coalescence of microcracks along α' -laths in the partial martensitic AM structure.

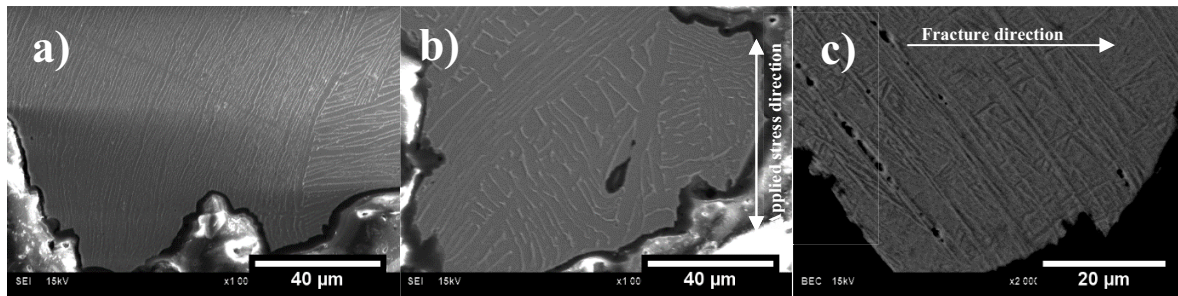


Figure 9. SEM micrographs of fracture cross-sections a) wrought, b) IC, and c) AM tensile test specimens

The fracture surfaces of the wrought, IC and AM are shown in **Figure 10**. The wrought and AM fracture surfaces (**Figure 10 a**) and **c**), respectively) showed relatively rougher surfaces, while the IC fracture surface appeared to be almost flat (**Figure 10 b**).

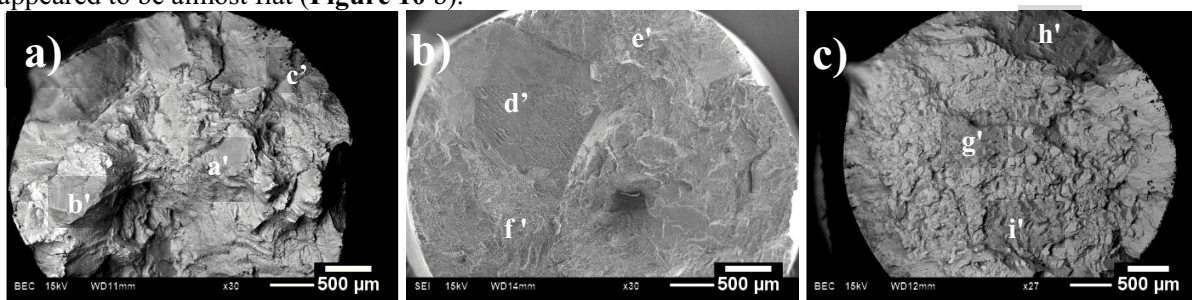


Figure 10. The fracture surfaces of a) wrought, b) IC, and c) AM Ti6Al4V

Figure 11 gives a higher magnification of the numbered areas in **Figure 10**.

The fracture surfaces of a specific area as shown in **Figure 10** of the wrought, IC and AM Ti6Al4V are given in Figure 11 a)-c), Figure 11 e)-f) and Figure 11 g)-i), respectively. The wrought and IC fracture surfaces revealed transgranular quasi-cleavage surface areas (larger in IC fracture) with ridges, microvoids

and laths. However, transgranular quasi-cleavage surface areas were relatively small (size of grains) in the AM fracture surface.

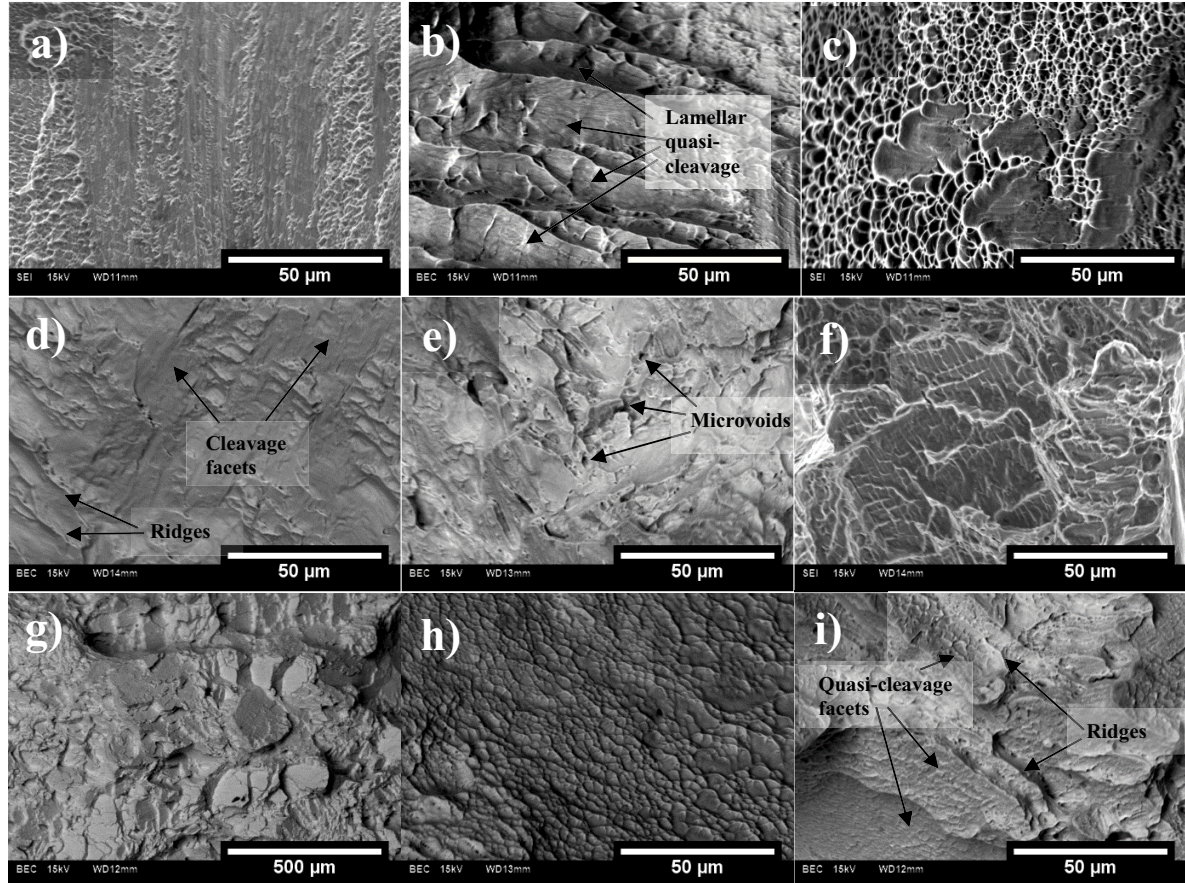


Figure 11. Fracture surfaces of a)-c) Wrought, d)-f) IC and g)-i) AM Ti6Al4V tensile specimens

4. Discussion

The columnar grains of the additive manufactured (AM) microstructure were oriented in the building direction (Y direction, see Figure 4 c) and crossing multiple-layer bands. The grain growth direction is counter to the heat flux. Since heat rapidly dissipates into the substrate (horizontally) when the process starts, forcing the grains to grow vertical [5]. The layer bands are the consecutive horizontal lines in Figure 4 c, the lines are visible due to the re-melting of previously solidified layers when a new layer is added. The re-melting also allows for epitaxial grain growth [8]. A change in the geometry of the grains in the top layers is visible in Figure 4 c. The grains change from columnar to coarse equiaxed grains, this is due to a slower cooling rate caused by the additional heat of the previous layers [9]. As the number of layers increases the heat inside the chamber rises, resulting in slower solidification of the top layers.

The smaller grains in the wrought microstructure led to a slight increase in hardness over the IC Ti6Al4V hardness [5],[10],[11]. However, the AM Ti6Al4V with partial martensitic microstructures proved to be superior in hardness. The high hardness of AM specimens is attributed to the presence of partial martensitic structure due to the high cooling rate during the 3D printing process [12].

The microstructure of Ti6Al4V influences the tensile properties, hardness and fracture modes of Ti6Al4V components. Depending on the manufacturing processes, Ti6Al4V can be of equiaxed, lamellar or martensitic microstructures. The martensitic microstructure showed high YS, UTS and hardness. While lamellar structures have a higher ductility and resistance to crack propagation.

When grain sizes decrease the number of grains in the same area increase, thus causing an increase in the number of grain boundaries. Grain boundaries are obstacles for a fracture movement causing entanglement and resulting in increased material strength [13]. This is in agreement following the comparison of microstructures and YS for IC and wrought Ti6Al4V tensile specimens. The low AM elongation is an indication of a brittle fracture mode, this was confirmed by Figure 8 c indicating a brittle fracture with no necking or permanent deformation. The Hall-Petch equation (1) indicates that smaller grain sizes promote higher YS and UTS [10].

$$\sigma_y = \sigma_0 + \frac{K_y}{\sqrt{d}} \quad (1)$$

Where parameter σ_y is the yield strength, σ_0 material constant, K_y strengthening coefficient and d the diameter of the average grain.

The fracture started with coalescence of microvoids, opening small pores between grain boundaries, or at any defect inside the specimen. The fracture propagates with increased applied stress. A 45° fracture deflection is observed towards the edge of the specimen (Figure 8) at the maximum shear stress (shear lips), this fracture segment is transgranular. Transgranular fractures are usually caused by catastrophic failure. Catastrophic failure occurs when necking or fracture already started, and the remaining intact specimen's area is reduced, thus decreasing the applied stress required for failure. The failure is sudden, fast and complete, cutting directly through grains.

The wrought manufactured specimen's overall fractured surface (Figure 8 c) resembles a partial inter-crystalline fracture, where some fractures occur between grains [14]. The IC fracture showed cleavage facets, microvoids and ridges on cleavage facets, resulting in an overall quasi-cleavage fracture. The quasi-cleavage fracture is common in high strength materials [14]. Most of the fracture surface of AM was observed as quasi-cleavage facets of a brittle nature. Secondary cracks as seen in Figure 9 c can only follow grains or lamella, and never be catastrophic [15], often corresponding to microvoid coalescence (MVC) [16].

5. Conclusions

The microstructure, hardness, tensile properties were investigated for additively manufactured (AM), investment casting (IC) and wrought Ti6Al4V alloy rods. The following conclusions were drawn:

- The investment cast Ti6Al4V had a colony lamellar structure in large equiaxed grains. The additively manufactured Ti6Al4V had long columnar grains with a Widmanstätten α' martensitic laths and retained β -grain boundaries. While the wrought Ti6Al4V microstructure was observed as smaller equiaxed grains with large colonies of fine lamellar and transformed β .
- The additive manufactured Ti6Al4V alloy specimen proved to be the hardest. The investment cast and wrought Ti6Al4V alloy specimens had similar hardness.
- The additively manufactured's martensitic microstructure led to a higher ultimate tensile strength and yielding strength, compared to investment cast and wrought Ti6Al4V. However, additively manufactured Ti6Al4V gave the lowest percentage elongation. The tensile properties of investment cast and wrought Ti6Al4V were similar.
- The investment cast fractures exhibited cleavage facets and microvoids, leading to a quasi-cleavage fracture. The additively manufactured fractures showed areas of intergranular fracturing and a large area of quasi-cleavage fracture. Wrought manufactured tensile tested specimens resulted in fracture

areas of quasi-cleavage and lamellar cleavage patterns. Thus, wrought, investment cast and additively manufactured Ti6Al4V revealed a typically brittle failure.

6. References

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