The Effect of Heat Treatments on Microstructure and Mechanical Properties of As-Extruded Ti-6Al-4V Alloy Rod from Blended Elemental Powders

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Abstract. In this study, Ti-6Al-4V bars were first prepared by extrusion of powder compacts from blended powder mixtures in the beta phase region, then the as-extruded bars were heat-treated following four different conditions: beta quenching and aging (βQA), broken up structure (BUS) treatment, solution treatment and aging (STA) and recrystallization annealing (RA). The effect of the heat treatments on microstructure and mechanical properties was studied using optical microscopy, scanning electron microscopy, and mechanical test to determine which heat treatment condition has the greatest impact on the mechanical properties of the as-extruded Ti-6Al-4V alloy. The results show that the as-extruded condition has the best balance of strength (1120 MPa of UTS) and ductility (11% of elongation to failure). βQA and STA lead to a slight increase in strength but ductility decreases considerably. After BUS and RA treatments, both strength and ductility are reduced. The relationship between processing, microstructure and properties was studied, and their implications towards fatigue behaviour and fracture toughness discussed.

Introduction

Powder metallurgy (PM) of titanium and its alloys is making its way as a reliable route to reduce the cost of final parts because of two main aspects. First, PM can be used as a near-net shape technique, saving time and material as it reduces the need of machining complex parts. Second, it can reduce the overall cost of the production after the metal extraction stage. The low-cost approach, known as blended-elemental (BE), is based on using cheap hydride-dehydride (HDH) titanium powder which is mixed with alloying powders. This blend is then pressed and sintered (P&S), taking advantage of the low strength and irregular shape of the powders. Even though its composition is usually not as pure as its wrought counterpart, a good balance of properties is still achieved: 985 MPa of ultimate tensile strength (UTS) and 8.8% of elongation in a 98% dense as-sintered Ti-6Al-4V with 0.24 wt.% of O and 0.1 wt.% of Cl [1].

P&S of BE powders usually yields relative densities around 95% and this leads to mechanical properties somewhat lower with respect to ingot metallurgy (IM) of Ti alloys [2]. One route to increase the final density is to design carefully the process: sieving the powders, using warm compaction or modifying the sintering parameters. Although fully-dense material are not achieved, the porosity is reduced enough to obtain satisfying properties. The other way to improve the mechanical properties is to use post-consolidation processes such as hot isostatic pressing (HIP) or thermomechanical processing to reduce the porosity with the assistance of pressure or plastic deformation. This usually results into nearly fully-dense materials that are competitive with IM.

Thermomechanical processing of α+β alloys includes hot forging, extruding or rolling in the β or α+β region. The sintered billet acts as a low-cost preform and the hot working brings the final shape and the full densification of the material, improving its mechanical properties [3, 4]. Thermomechanical processing can also be applied to press compacts without previous sintering stage, consolidating the material during the thermomechanical processing, being able to achieve mechanical properties similar to thermomechanically processed alloys previously sintered [5, 6].

To further improve the mechanical properties of PM Ti alloys, heat treatments can be applied to tailor the microstructure and the resulting mechanical properties. This has proven successful for both pre-alloyed (PA) and BE approaches for Ti-6Al-4V, using HIP and thermomechanical processing. Broken-up structure (BUS) treatment has been applied to PA and BE fully-dense HIP billets resulting in high strength and ductility for both routes (1000 MPa of UTS and 9% of elongation for BE and 1000 MPa of UTS and 15% of elongation for PA) [7-9]. Another heat treatment that yields high strength and moderate ductility for BE HIP billets is the solution and aging treatment (STA) (1190 MPa of UTS and 10% of elongation) [9]. The processes that combine thermomechanical processing and heat treatments generally involve deforming the material in the α+β region, as it can give room to a wider selection of microstructures due to the recrystallization of the deformed α phase. The typical heat treatments in this case are annealing treatments like recrystallization annealing (RA) and α+β annealing and aging. These were applied to a BE Ti-6Al-4V sheet rolled in the α+β region, resulting in increased tensile strength and ductility compared to that of the sintered and β annealed [1].

In this work, a selection of heat treatments was applied to as-extruded Ti-6Al-4V alloy rods in the β region from sintering of BE powders and their effect on microstructure and mechanical properties was studied. The aim is to find the best conditions that should translate into improved fatigue performance or, conversely, high fracture toughness.

Experimental Procedure

HDH Ti powders and 60 wt.% Al-40 wt.% V master alloy powders were mixed in a V-mixer to get a homogeneous blend. Cylindrical compacts of 56 mm of diameter were produced using warm uniaxial compaction in air at 230 °C and applying a pressure of 370 MPa. The compacts were vacuum sintered at 1280 °C for 2 hours, with a vacuum level of 10^{-1} Pa. The vacuum sintered billets were heated up to 1150 °C using an induction furnace in air and immediately transferred to a horizontal 300-Ton press for extrusion to produce Ti-6Al-4V alloy bars using an extrusion ratio of ~ 7.5. The extruded bars were heat-treated following different procedures: β-quenching and aging (βQA), BUS, STA and RA. The details of the heat treatments studied are shown in Table 1.

Table 1. Details of the heat treatments studied.

Heat treatment	First stage	Cooling	Second stage
βQA	1050 °C, 1h	WQ	600 °C, 2 h
BUS	1050 °C, 1h	WQ	805 °C, 24 h
STA	950 °C, 1h	WQ	540 °C, 6 h
RA	925 °C, 4h	50 °C/h	760 °C, no holding

Legend: βQA (beta quenching and aging), BUS (broken-up structure), STA (solution treatment and aging), RA (recrystallization annealing), WQ (water quenching).

The density of the green compacts was determined by mass and volume measurements, while for the as-sintered and as-extruded material it was measured using Archimedes' method. The microstructure and tensile properties of the as-extruded and heat-treated bars were studied using optical microscopy (OM) and scanning electron microscopy (SEM, Hitachi S-4700) and mechanical test. In order to perform microstructure observation, cold-mounted samples were ground and polished using diamond suspension and silica suspensions, respectively, and finally etched using Kroll's reagent. Dog-bone shaped specimens, with a gage length of 20 mm and a cross-section of 2 x 2 mm^2 were used for tensile testing, which was performed using a 4202 Instron universal testing machine with a 5 kN load cell and an extensometer.

Results and Discussion

Table 2 shows the relative densities of green compact, sintered material and extruded material. The green and sintered density are high for 500g-compacts, which proves the efficacy of the warm compaction and vacuum sintering parameters. After extrusion, the density is almost the theoretical value, so the material can be considered fully-dense. However, some residual pores are still present.

Table 2. Relative densities of green compact, as-sintered and as-extruded Ti-6Al-4V.

Property	**Green compact**	**As-sintered**	**As-extruded**
Relative density [%]	84.04 ± 1.45	96.76 ± 0.47	99.99 ± 0.12

Fig. 1 shows optical micrographs of the as-extruded Ti-6Al-4V alloy in the transverse and longitudinal directions. Only a small amount of pores is left after extrusion and most of them are elongated, needle-like pores along the extrusion direction. After image analysis of the pores, three groups are present: (1) small, round pores with size of smaller than 2 μm, (2) needle-like pores with dimensions no larger than 10 μm of width and 50 μm of length, and (3) big round pores with diameter no larger than 60 μm. This last group is usually located at the centre of the cross section of the bar, due to the lower amount of plastic deformation experienced by the material, and is the one that will have the larger impact on the mechanical properties.

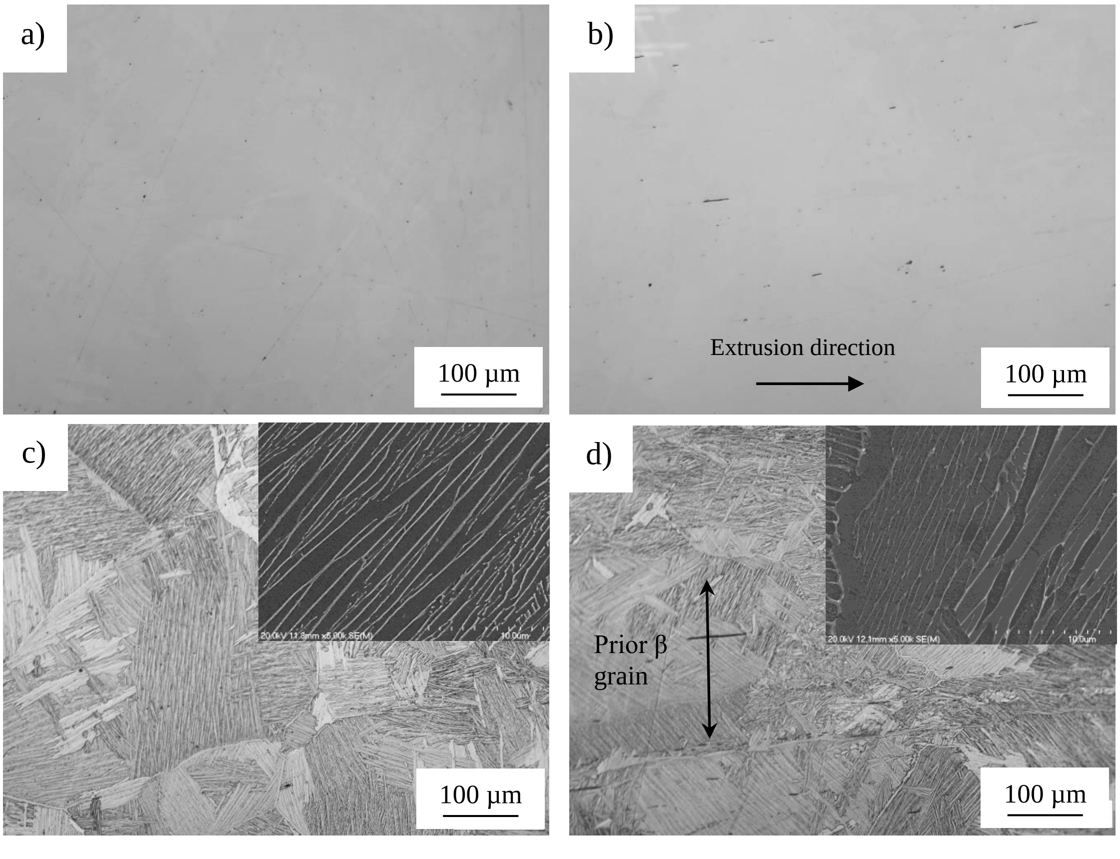

Fig. 1. Optical micrographs of the as-extruded Ti-6Al-4V alloy: transverse section before (a) and after (c) etching, and longitudinal section before (b) and after (d) etching. For (c) and (d) SEM micrographs are presented as inset.

The microstructure of the as-extruded Ti-6Al-4V alloy consists of very fine lamellar colonies (Fig. 1). The average width of the lamellae is 1.2 μm and their average length is 28 μm. The size of the colony is in the order of 100 μm. There is not much difference in the microstructure of the transverse and longitudinal sections, apart from the presence of heavily deformed prior β grains along the extrusion direction.

Fig. 2 shows the microstructure of the extruded Ti-6Al-4V bars after heat treatment. After βQA, the microstructure is composed of very fine acicular α, of an average width of 0.5 μm (Fig. 2a) whereas a similar microstructure of coarser α laths, with an average width of 2.5 μm, are obtained via the BUS treatment (Fig. 2b). The higher heat treatment temperature and longer holding time of this step allows to decompose all the α' martensite into plates that have a greater thickness than that of the as-extruded condition. STA leads to a mixed microstructure formed by primary α plates (present after quenching from 955 °C) usually grouped in small colonies, that are separated by the regions of extremely fine acicular α formed after the decomposition of α' martensite (Fig. 2c). In this case, the thickness of the primary α lamellae is of a similar value to the thickness of the phase that separates them, which is an average of 2 μm. After RA, the microstructure stays as lamellar with no equiaxed α, meaning that recrystallization did not happen during the RA process, as the extrusion was performed on the β field. The average thickness of α lamellae is of 3.5 μm and the size of the sub-colonies is about 15 μm (Fig. 2d).

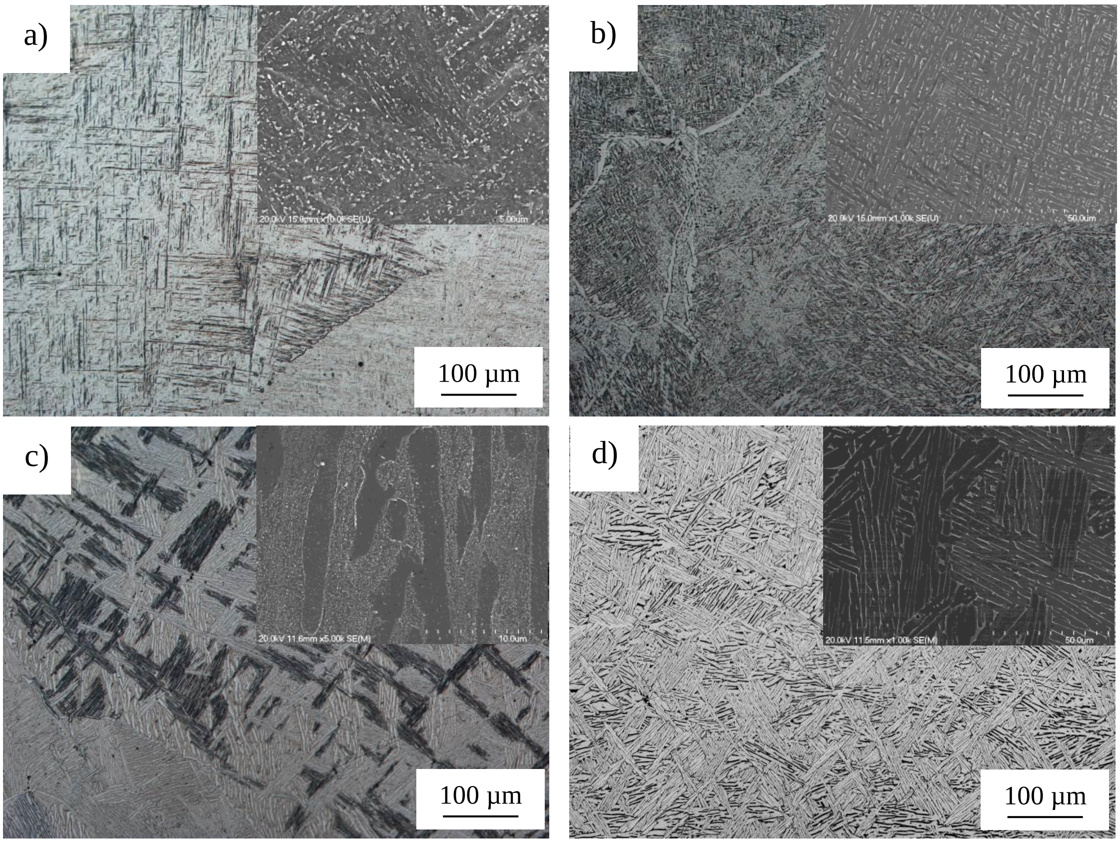

Fig. 2. Optical and SEM (inset) micrographs of the extruded and heat-treated Ti-6Al-4V alloy after various heat treatments: a) βQA, b) BUS, c) STA, and d) RA.

Table 3 shows the tensile properties (yield strength-YS, UTS and ductility) of the materials studied. The strength of the Ti-6Al-4V alloys in each condition satisfies the standard requirements for IM Ti-6Al-4V, while for the ductility only the as-extruded and BUS conditions satisfy the standard. The as-extruded Ti-6Al-4V shows intermediate-to-low strength and the highest ductility values compared to that of the heat-treated bars. However, the strength level achieved in the as-extruded condition is higher than that of other PM Ti-6Al-4V [9]. BUS and RA achieve higher ductility than βQA and STA as these treatments coarsen the microstructure and lead to lower strengths with respect to that of the as-extruded condition. Comparing the properties of BUS treatment in this work with literature values of the same heat treatment in fully-dense BE Ti-6Al-4V [9], the Ti-6Al-4V alloy obtained via the process discussed here is stronger (1080 MPa of UTS compared to 1000 MPa) and slightly more ductile (10% of elongation compared to 9%).

Table 3. Tensile properties of the extruded and heat-treated Ti-6Al-4V alloy.

Condition	YS [MPa]	UTS [MPa]	Ductility [%]
As-extruded	976 ± 6	1118 ± 7	10.8 ± 0.2
βQA	1077 ± 6	1162 ± 17	8.6 ± 2.1
BUS	997 ± 6	1084 ± 9	10.3 ± 2.4
STA	1102 ± 32	1169 ± 27	5.7 ± 1.8
RA	943 ± 12	1051 ± 13	9.0 ± 1.8
ASTM Standard (Grade 5) [10]	828	895	10

The heat treatments that result in very fine acicular α (βQA and STA) yield the highest strength and the lowest ductility values. A comparison of the properties of the STA in this work with other fully-dense BE alloy [9] indicates that both of them have similar strengths (1170 MPa and 1180 MPa of UTS) but lower ductility (6% compared to 10% of elongation, probably due to the higher oxygen content).

To further understand the relationship between the microstructure and the tensile properties, the yield strength was plotted against an inverse of the slip length, which is estimated as the average width of the α plates, to represent the Hall-Petch relationship (Fig. 3). Most of the conditions follow a linear relationship, which proves the strengthening effect of refining the microstructure. The only case that does not follow the rule is the STA treatment, where D was taken as the width of the primary α plates. In this case, strength is controlled by another microstructural parameter, for instance the size of the martensite or α plates within the other phase.

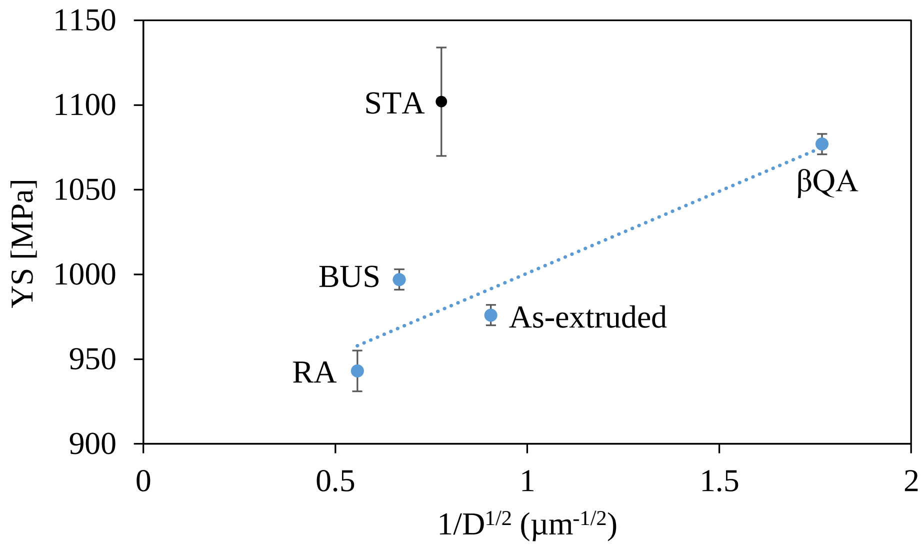

Fig. 3. Hall-Petch relationship between yield strength (YS) and α lamellae width (D) of the extruded and heat-treated Ti-6Al-4V alloy.

The relationship between ductility and the width of α plates is plotted in Fig. 4. Again, all conditions follow a trend except for the case of STA treatment. Due to the large standard deviation of the ductility values, there is little dependence on the microstructural features. However, the as-extruded condition, with its fine lamellar microstructure, shows a higher ductility than the conditions that present martensite in their microstructure (STA and βQA). A similar behaviour was found when studying the relationship between cooling rate from the β phase and elongation and the reason for the presence of this maximum is the change of the fracture mode [11]. The extruded bars were left to cool down in air from the β phase, which is a suitable cooling rate to obtain fine lamellar microstructures.

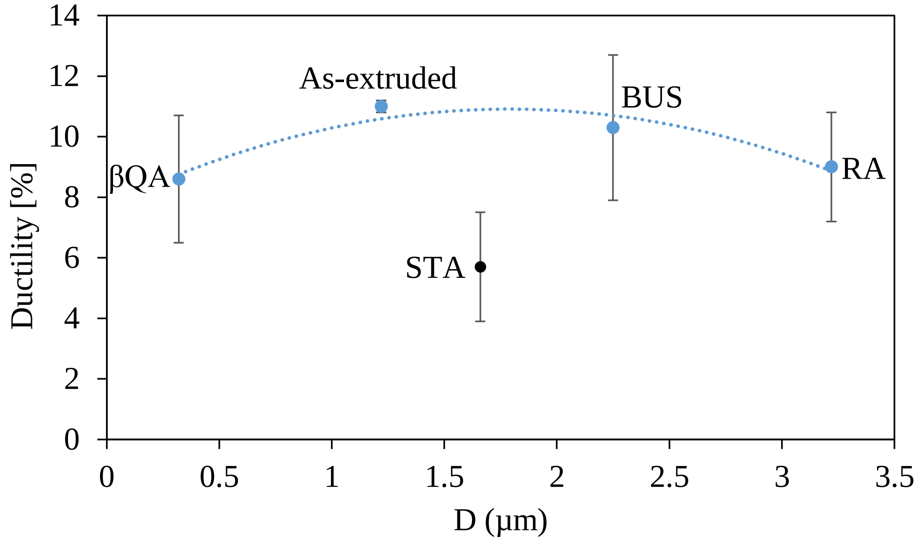

Fig. 4. Relationship between the ductility and the average width of α plates of the extruded and heat-treated Ti-6Al-4V alloy.

From the literature, if the lamellae are forming colonies as the ones seen in the as-extruded or RA microstructures, the virtual slip length used for the Hall-Petch relationship is the size of this colony, so microstructures where the α plates are arbitrarily distributed have improved properties [9, 11]. In this work, the presence or not of α colonies is not affecting the mechanical properties, as BUS (which does not have a colony microstructure) follows the same trend of tensile properties with the α width found for the as-extruded or RA conditions (where the microstructure shows colony substructures). A study using neural networks to model the tensile properties of Ti-6Al-4V from various microstructural parameters found that the most controlling parameter is the α lath thickness, more than the size of the colony [12].

All the microstructures developed in this work are based on lamellar microstructures or decomposed martensite, missing microstructures that have equiaxed grains as one of their constituents (full equiaxed or bi-modal microstructures). Considering this, microstructural tailoring is not a route for improving the properties of the as-extruded PM Ti-6Al-4V bars processed in this work. The as-extruded material satisfies the standard requirements for the wrought alloy and it has one of the highest ductilities studied, while keeping an UTS comparable to the solution treated and aged conditions. In order to improve the ductility of the extruded PM Ti-6Al-4V, other routes like modifying the extrusion temperature (to obtain equiaxed grains), reducing the overall oxygen content or the pore size could be investigated.

Considering high cycle fatigue behaviour, the most important predicting parameter for the life of the material is its UTS, as it is related to the point where the crack initiates. Therefore, very fine microstructures and high UTS are preferred. For increasing fracture toughness, coarse microstructures and lower yield strengths are preferred, as they reduce the actual stress concentration along the crack propagation line. The as-extruded condition shows a promising behaviour for both fatigue and fracture properties, because compared to the other conditions studied it has high UTS while its YS is low. This way, crack initiation is prevented and the tip of the crack is more blunted by the bigger plastic zone. βQA and STA treatments, because they display high UTS and YS, are more likely to display higher fatigue strengths but lower fracture toughness. In the case of BUS and RA treatments, it is expected that their fatigue strength will be lower but their fracture toughness will be higher compared to the as-extruded alloy.

Conclusions

From this study about the effect of different heat treatments on the properties of PM blended elemental Ti-6Al-4V extruded rods it is found that the as-extruded material presents the best combination of strength and ductility, with 1120 MPa of UTS and 11% of elongation to fracture. The microstructural tailoring of the different heat treatments is able to improve the tensile strength, in the case of βQA and STA treatments, but not the ductility. It is found that the key parameter to control the tensile properties is the thickness of the α laths. Strength increases when the thickness decreases but the ductility remained constant regardless of the size of the laths.

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