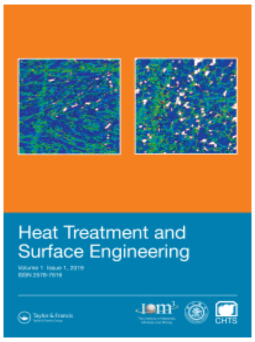

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RESEARCH ARTICLE

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Effect of $\alpha+\beta$ solution treatment and aging on the performance of powder forged Ti-5Al-2.5Fe

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ABSTRACT

Amongst titanium alloys, the $\alpha+\beta$ Ti alloys are the ones with the best compromise between strength and fracture toughness. However, the applicability of $\alpha+\beta$ Ti alloys is limited by their higher cost compared to other structural metals. Reduction of the cost can be obtained using cheaper alloying elements and creative manufacturing processes. This study aims to investigate if $\alpha+\beta$ Ti alloys can be produced at lower cost using powder forging and to understand the effect that the post-forging $\alpha+\beta$ solution treatment and aging have on the microstructure and mechanical properties. We demonstrate that the proposed approach is a viable alternative to manufacture $\alpha+\beta$ Ti alloys and that the chosen forging temperature (i.e. thermal history) is critical for producing high-strength ductile alloys. The microstructural modification induced by the $\alpha+\beta$ solution treatment and aging generally increases both the strength and the ductility, but the actual improvement is still dictated by the conditions used to thermomechanically deform the $\alpha+\beta$ Ti alloy.

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Introduction

The wider adoption of titanium and its alloys in engineering applications is held up by its higher extraction and manufacturing costs with respect to other structural metals like steel and aluminum alloys [1,2]. However, Ti alloys are preferred over other structural metals [3,4] where low density, high strength, corrosion resistance, and biocompatibility, or high temperature strength, are simultaneously required [5,6]. The wide range of properties achievable in Ti alloys is principally connected to the fact that Ti has two allotropes, and the addition of different alloying elements can be used to tailor the microstructure. More in detail, at room temperature pure Ti has a hexagonal close-packed lattice, known as α, which transforms into a body-centered cubic lattice (i.e. β) once a critical temperature is overcome. The latter is called β transus and in the case of pure Ti is 882°C [7]. Chemical elements that increase the β transus are called α-stabilizers, where the main element used is Al, whilst elements that lower the β transus are called β-stabilizers. The addition of either type of alloying elements creates a two-phase region where the α-Ti and β-Ti phases coexist. This opens up the possibility to have, at room temperature, Ti alloys characterized by a microstructure composed of only α-Ti or only β-Ti grains, respectively labeled as α and β Ti alloys, or by mixture of the two phases, known as $\alpha+\beta$ Ti alloys [8].

Among Ti alloys, $\alpha+\beta$ alloys are the most extensively used as they provide the best compromise between strength and toughness. Amongst $\alpha+\beta$ alloys, Ti-6Al-4V is the most widely studied and employed. However, V is more expensive than Ti, which increases the cost of the material, and the release of V ions has been reported to be cytotoxic [9], which is relevant for biomedical applications [10]. To tackle the cytotoxicity problem, V-free $\alpha+\beta$ alloys such as Ti-6Al-7Nb [11,12] and Ti-5Al-2.5Fe [13–15] as well as β alloys like Ti-13Nb-13Zr [16,17] were thus proposed and commercially developed as the second generation of Ti alloys for biomedical applications. Both Ti-6Al-7Nb and Ti-5Al-2.5Fe have mechanical performance equivalent to Ti-6Al-4V but Ti-5Al-2.5Fe has the added advantage of being cheaper due to the inclusion of Fe rather than V or Nb in the chemical composition. Reduction of the intrinsic cost of Ti and of its manufacturing costs through the use of alternative processes like powder metallurgy has, actually, been identified as key aspect to increase the deployability of Ti alloys in diverse engineering applications [18–20].

Demonstration of the possibility of manufacturing the Ti-5Al-2.5Fe alloy by means of the blending elemental approach and the analysis of the microstructural evolution with the sintering parameters was done by Siqueira et al. [13,14]. In order to further reduce the manufacturing costs associated with the processing of the Ti-5Al-2.5Fe alloy through powder metallurgy, Jia et al. [15] proposed the use of induction sintering rather than vacuum sintering in electrical resistance furnaces demonstrating that the consolidation time

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can be shortened from hours to minutes. Processing of the Ti-5Al-2.5Fe alloy using metal injection moulding and the properties of the sintered alloy were quantified by Xu and Nomura [21,22]. Regarding advanced powder metallurgy techniques, Yamanoglu et al. [23] used hot pressing to obtain almost fully-dense Ti-5Al-2.5Fe samples from mechanically alloyed powders whereas Hagiwara et al. [24] used hot isotactic pressing to reduce the residual porosity and increase the mechanical properties [25].

The Ti-5Al-2.5Fe alloy is a cheaper option with equivalent mechanical properties to Ti-6Al-4V and its processing via powder metallurgy can further reduce its cost. Both conventional and advanced powder metallurgy techniques have been investigated to produce the Ti-5Al-2.5Fe alloy where the latter generally permits to achieve better mechanical behavior. However, the processing of the powder metallurgy Ti-5Al-2.5Fe alloy by means of thermomechanical processing has not been investigated in detail, especially when coupled with a subsequent heat treatment designed to improve the mechanical performance. The readers is referred to the following references for a comprehensive understanding of the effect of thermomechanical processes on Ti alloys [26,27]. Therefore, the aim of this study is to investigate the effect that $\alpha+\beta$ solution plus aging heat treatments have on the microstructural evolution and mechanical properties of the Ti-5Al-2.5Fe alloy obtained by means of the blended elemental approach and further densified through the thermomechanical plastic deformation process of hot forging.

Experimental procedure

The raw materials used to produce the Ti-5Al-2.5Fe alloy using the blended elemental approach were hydride-dehydride Ti (angular, <75 µm, oxygen = 0.27%), atomized Al (spherical, <45 µm, oxygen = 0.56%), and carbonyl Fe (dendritic, <10 µm, oxygen = 0.32%) powders. The correct amount of the three elemental powders was blended by means of a two-roll mill run for 24 h to ensure homogeneity, cold uniaxially pressed into 56 mm diameter samples using 400 MPa of pressure, and vacuum sintered at 1250°C for 2 h using a vacuum level better than 10^{-3} Pa and 10°C/min as heating and cooling rates, which are common sintering conditions for blending elemental powder metallurgy Ti alloys [28]. From Figure 1, which shows the microstructure of the as-sintered Ti-5Al-2.5Fe alloy, it can be seen that the material is characterized by the typical lamellar microstructure of slow-cooled $\alpha+\beta$ Ti alloys as well as primarily spherical residual pores (approx. 8%).

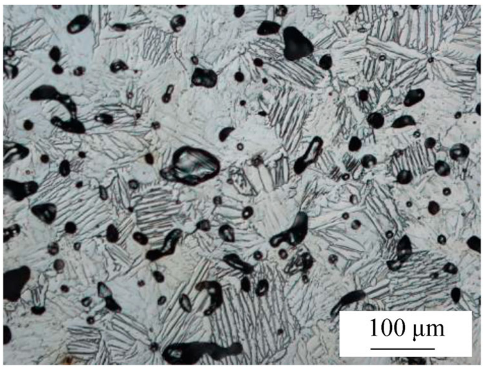

Figure 1. Micrographs of Ti-5Al-2.5Fe alloy sintered at 1250°C for 2 h.

The sintered samples were subsequently forged either in the β field at 1250°C or in the $\alpha+\beta$ field at 950°C using a height forging ratio of 2:1. Subsequently, the forged samples were heated to 950°C for 2 h, quenched in water, and finally aged at 550°C. The aging times of the $\alpha+\beta$ solution plus aging heat treatment analysed included 2, 4, 6, 8, and 24 h.

The forged and heat-treated Ti-5Al-2.5Fe samples were prepared for metallographic analysis using the classical SiC grinding and OPS (oxide polishing suspension) route. A distilled H_2O-based solution comprising 2 ml of HF and 6 ml of HNO_3 was used to chemical etch the samples to reveal the phases. An Olympus BX60 optical microscope was used to metallographically analyse the samples. A Philips X'Pert equipment operated at 45 kV and 40 mA was used to obtain the XRD (X-ray diffraction) patterns of the forged samples in the 30° to 80° scanning angle using a step of 0.013°. The tensile behavior of the forged and heat-treated Ti-5Al-2.5Fe samples was quantified using dog-bone specimens with rectangular cross section of 2 mm × 2 mm and gage length of 20 mm. Samples were tested at a cross-head speed of 0.1 mm/min using an Instron 33R4204 universal testing machine equipped with a mechanical extensometer. A minimum of three samples for each condition was tested in order to calculate the average tensile properties.

Results and discussion

The results of the microstructural analysis and of the identification of the phases of the Ti-5Al-2.5Fe alloy forged at either 1250°C or at 950°C are reported in Figure 2. The microstructure of the alloy forged at 1250°C is the typical lamellar structure of $\alpha+\beta$ Ti alloys cooled from within the β field. It is worth noticing that the interlamellar spacing is small (i.e. very fine $\alpha+\beta$ lamellae), the size of the $\alpha+\beta$ colonies is relatively coarse (in the range of hundredth of µm), and some very few small, isolated pores are present, making the alloy fully dense (Figure 2(a)). This microstructure is the result of the thermal history of the alloy, which was originally vacuum sintered at 1250°C and, therefore, characterized by a coarse lamellar

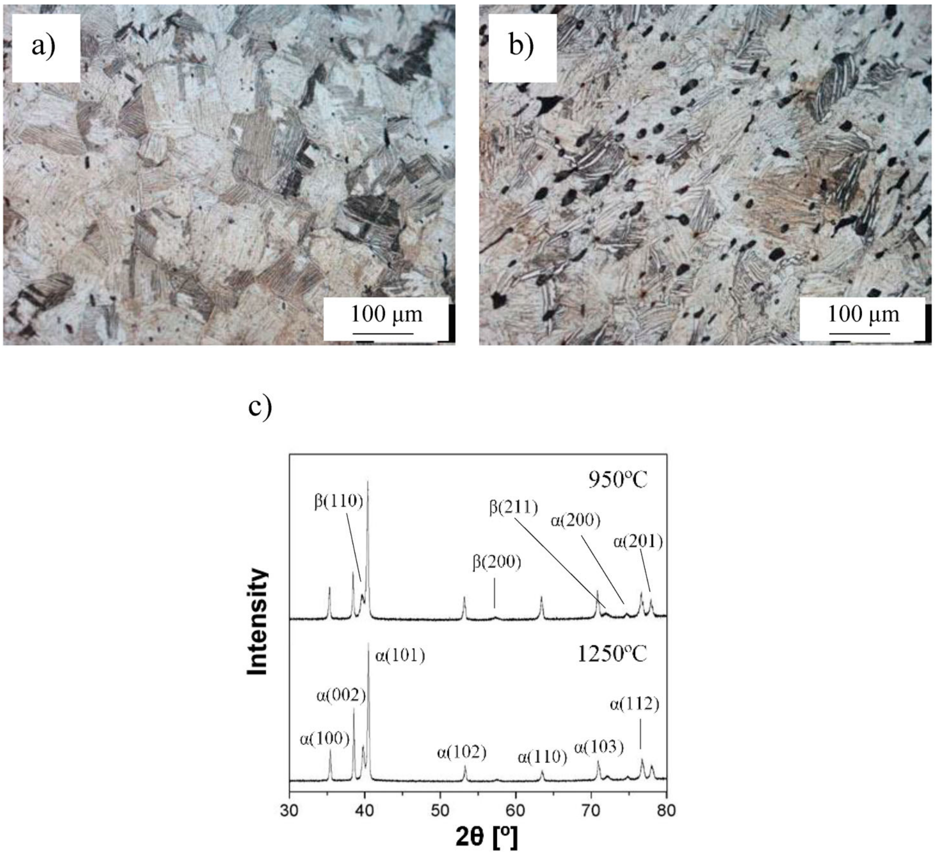

Figure 2. Microstructural analysis of the Ti-5Al-2.5Fe alloy: (a) forged at 1250°C, and (b) forged 950°C; and (c) XRD patterns.

structure and an approximate amount of residual porosity of 8%, which is characteristic of sintered Ti alloys [29,30]. Heating to 1250°C for forging did not significantly increase the size of the *α*+*β* colonies with respect to the sintered alloy, but the fast cooling rate of hot forging in air led to a remarkable refinement of the *α*+*β* lamellae. The high deformability of the alloy while having an equiaxed *β* grains microstructure during forging permitted to fully seal the residual porosity [31,32].

The microstructure of the alloy forged at 950°C looks like a lamellar structure, but it is also characterized by the presence of a small amount of elongated primary *α* grains, more typical of a bimodal microstructure. The interlamellar spacing is greater (i.e. coarser *α*+*β* lamellae), but the size of the *α*+*β* colonies slightly smaller, with respect to the alloys forged at 1250°C, and some residual porosity (∼4%) is still present (Figure 2(b)). The presence of the primary *α* grains, the smaller colonies size, and the coarser lamellar structure are due to the fact that the forging temperature of 950°C sits at the upper limit of the *α*+*β* field [33,34]. Consequently, less time is available for grain growth, not the whole alloy transforms into equiaxed *β* grains, and cooling happens from a lower temperature. Due to the lower forging temperature, the alloy is also characterized by lower deformability, and the application of the plastic deformation through forging is not able to complete seal the residual porosity, although its amount is reduced approximately by half. Coherently with the results of the microstructural analysis, the XRD patterns of (Figure 2(c)) show that, regardless of the forging temperature, the alloy is composed by the *α*-Ti and *β*-Ti phases. However, the intensity of the peaks related to the *β*-Ti phase is stronger for the Ti-5Al-2.5Fe alloy forged at 1250°C compared to 950°C, which is consistent with the results of the microstructural analysis.

The microstructural evolution of the Ti-5Al-2.5Fe alloy forged at 1250°C with the aging time is shown in Figure 3. Generally, a coarser lamellar microstructure compared to the forged alloy and characterized by the presence of thick *α* grain boundaries is obtained, regardless of the aging time. However, the latter significantly affects the size of the *α*+*β* lamellae, the size of the *α* grain boundaries and, therefore, the relative amount of the *α*-Ti and *β*-Ti phases present in the microstructure. The effect of the aging treatment on the size of the *α*+*β* colonies is much less remarkable and aging does not affect the few pores present. The rapid cooling of the Ti-5Al-2.5Fe alloy during quenching leads to the formation of a Widmanstätten microstructure as the *β*-Ti phase present, which constitutes most of the Ti-5Al-2.5Fe alloy at 950°C, transforms displacively into martensite while the remaining of the *α* grain boundaries are not affected by quenching [35].

Upon the first heating to the aging temperature of 550°C, the martensite present decomposes into the

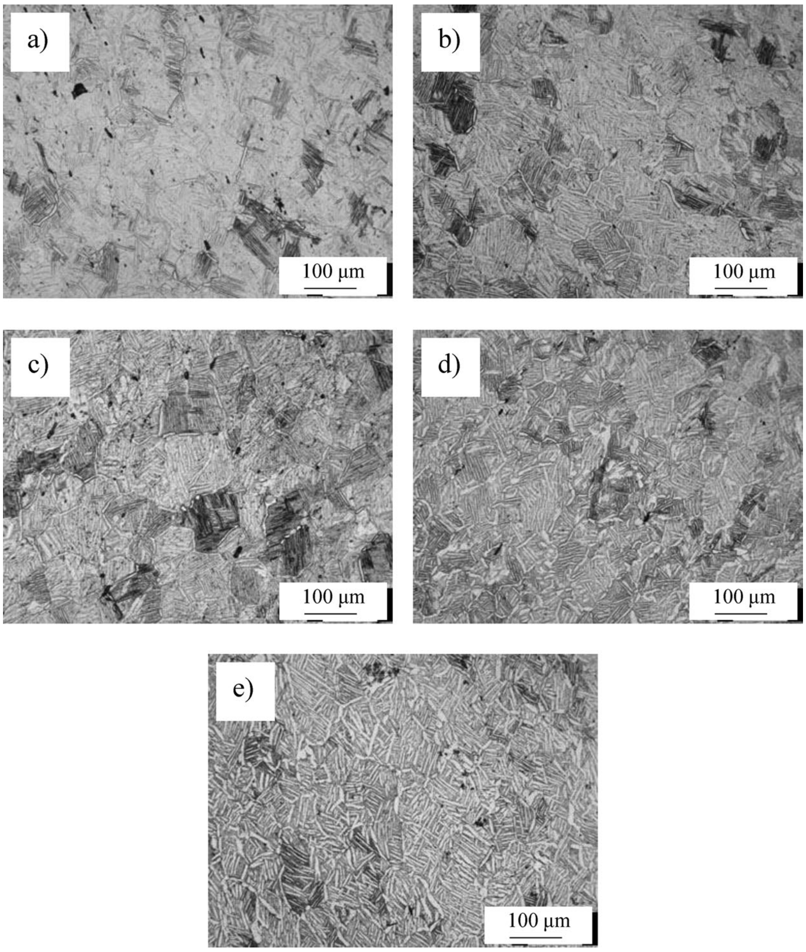

Figure 3. Microstructural evolution with the aging time of the Ti-5Al-2.5Fe alloy forged at 1250°C: (a) 2 h, (b) 4 h, (c) 6 h, (d) 8 h, and (e) 24 h.

stable α-Ti and β-Ti phases leading to the coarsening of the primary α grains and the formation of a coarse α+β lamellar structure (Figure 3(a)). As the aging time increases, the width of the α grain boundaries increases and so does the amount of α-Ti phase present at the expense of the β-Ti phase. This is due to the change in relative amount between the primary α grains and transformed β. Therefore, a much more marked difference is found between the Ti-5Al-2.5Fe alloy aged for 8 h (Figure 3(d)) compared to that of the alloy aged for 24 h (Figure 3(e)).

Figure 4 shows the mechanical properties of the Ti-5Al-2.5Fe alloy forged at 1250°C and from the representative stress–strain curves it can be seen that the forged alloy without and with heat treatment undergoes elastic and plastic deformation when subjected to a quasi-static uniaxial load (Figure 4(a)). The actual value at which the alloy transition from elastic to plastic deformation depends on the thermal history of the alloy [36], and so do the maximum strength and the elongation at fracture.

In particular, from (Figure 4(b)), where the average mechanical properties of the Ti-5Al-2.5Fe alloy forged at 1250°C are shown, the strength initially increases and then decreases with the aging heat treatment time whilst the ductility as the opposite trend. With

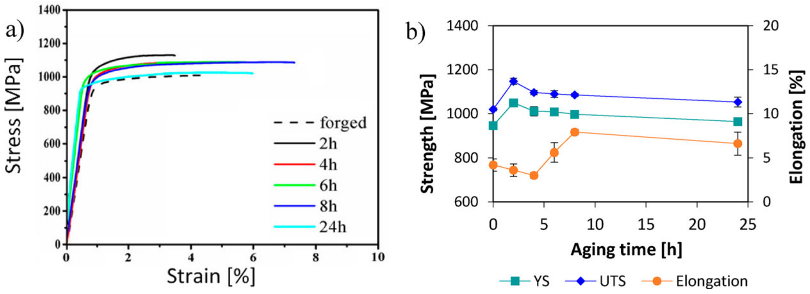

Figure 4. Mechanical properties of the Ti-5Al-2.5Fe alloy forged at 1250°C: (a) representative stress-strain curves, and (b) average mechanical properties. Note: the aging time of 0 h correspond to the forged alloy.

respect to the forged alloy, the highest gain in strength is obtained right after quenching of the samples through the initial aging treatment of 2 h, reaching YS and UTS of 1049 ± 11 and 1147 ± 14, respectively. This increment is due to the decomposition of the martensite formed upon quenching into the coarse lamellar microstructure [37,38] comprising α grain boundaries and coarse α+β lamellae (Figure 3). The coarsening of the microstructural features coupled with a greater amount of material transforming into the α-Ti phase induced by a longer aging heat treatment in the α+β field justifies the progressive decreasing of the strength. Consequently, the alloy aged for 24 h has the lowest strength among the heat-treated samples, which is, however, still slightly higher (on average 25 MPa) with respect to the forged alloy. Analysing the variation of the strain (Figure 4(b)), the initial transformation of the quenched microstructure during the early hours of the aging heat treatments leads to the decrement of the ductility in comparison to the forged alloy due to the nucleation and growth of the α-Ti phase at the grain boundaries [38,39]. Moreover, the coarsening of the microstructural features with the subsequent increment of the aging time actually permits to recover the ductility of the alloy reaching the highest strain value of 7.9 ± 0.3 after 8 h of aging heat treatment. However, the increase of the aging time to 24 h starts a decreasing trend of the ductility due to significant increment of the amount of α-Ti phase present in the microstructure (Figure 3(e)).

From Figure 5, which shows the microstructural evolution of the Ti-5Al-2.5Fe alloy forged at 950°C with the aging time, it can be seen that the aged Ti-5Al-2.5Fe alloy is characterized by a bimodal microstructure composed of primary α grains, some equiaxed and some as α grain boundaries, and very coarse transformed β structure. As for the Ti-5Al-2.5Fe alloy forged at 1250°C, the fast cooling imposed by quenching transforms the β-Ti phase into martensite. However, due to its thermal history, prior to quenching the Ti-5Al-2.5Fe alloy forged at 950°C has a much greater amount of primary α grains and, consequently, a lower amount of β-Ti phase that transforms martensitically. The effect of the initial aging on the decomposition of the martensitic phase into transformed β, as well as the effect of the aging time on the microstructural feature, is similar to that described for the Ti-5Al-2.5Fe alloy forged at 1250°C (Figure 3). Therefore, the aging heat treatment leads to the growth of the primary α grains/α grain boundaries coupled with the coarsening of the α+β lamellae (Figure 5(a)), where the overall coarsening progresses with the aging time. A significantly higher amount of primary α grains/α grain boundaries is, thus, found in the Ti-5Al-2.5Fe alloy forged at 950°C and aged for 24 h (Figure 5(e)) with respect to the same alloy aged for 8 h (Figure 5(f)).

Representative stress–strain curves as well as the average mechanical properties of the Ti-5Al-2.5Fe alloy forged at 950°C without and with the subsequent solution and aging heat treatment are shown in Figure 6. The forged and heat-treated Ti-5Al-2.5Fe alloy is always characterized by an elastoplastic behavior regardless of the processing conditions. However, as in the case of the Ti-5Al-2.5Fe alloy forged at 1250°C, the actual maximum strength and elongation at fracture values depend on the thermal history of the alloy (Figure 6(a)). Consequently, it can be seen that regardless of the heat treatment and the actual aging time, the Ti-5Al-2.5Fe alloy forged at 950°C always displays higher strength but lower strain values with respect to the Ti-5Al-2.5Fe alloy forged at 1250°C (Figure 4(a)).

Depending on the actual aging conditions, the strength is between 55 and 150 MPa higher in the Ti-5Al-2.5Fe alloy forged at 950°C but the ductility is between 0.7% and 2.8% lower (Figure 6(a)). This difference in average mechanical properties values is primarily related to the fact that the Ti-5Al-2.5Fe alloy forged at 950°C starts from a bimodal microstructure composed of elongated primary α grains, coarser α+β lamellae but finer α+β colonies, and residual porosity (Figure 2(b)). The presence of residual pores significantly affects by ductility of the alloy, which is always lower compared to the Ti-5Al-2.5Fe alloy forged at 1250°C, but not so much the strength as the negative effect of the residual pores is overcompensated by the difference in colony's size, lamellae's size, and greater amount of primary α grains/α grain boundaries. From the analysis of the average mechanical properties of the Ti-5Al-2.5Fe alloy forged at 950°C (Figure 6(b)), it can be seen that the overall trends of the variation of the strength and of the ductility are comparable to those of the Ti-5Al-2.5Fe alloy forged at 1250°C (Figure 4(b)), as both alloys undergo similar microstructural modification during the aging heat treatment. The strength progressively decreases with the aging time after having reached the highest value (YS of 1198 ± 31 and UTS of 1293 ± 37) after the initial aging of 2 h, as a consequence of the martensitic decomposition and coarsening of the microstructure. Coherently, the maximum strain initially decreases, then increase, and eventually starts to decrease again for a too-long aging heat treatment, as does the strength. However, the Ti-5Al-2.5Fe alloy forged at 950°C and overaged for 24 h has, on average, still better strength (~36 MPa) and ductility (4.5%) in comparison to the forged alloy.

The mechanical properties of the forged Ti-5Al-2.5Fe alloy without and with post-forging α+β solution treatment and aging are compared with relevant α+β Ti alloys produced using different metallurgical routes [7,24,40,41] in Figure 7. Generally, the forged and heat-treated Ti-5Al-2.5Fe alloy of this study has

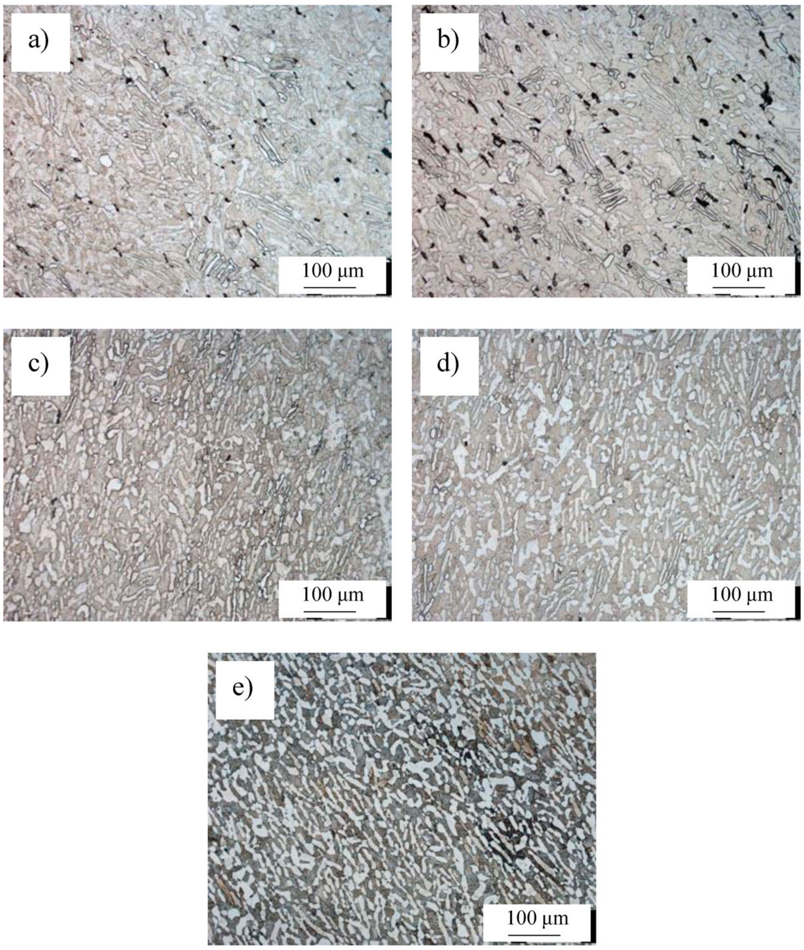

Figure 5. Microstructural evolution with the aging time of the Ti-5Al-2.5Fe alloy forged at 950°C: (a) 2 h, (b) 4 h, (c) 6 h, (d) 8 h, and (e) 24 h.

comparable or better YS compared to both wrought and powder metallurgy representative α+β Ti alloys. In particular, YS is higher than that of cast, sintered or hot isostatically pressed Ti-5Al-2.5Fe and Ti-6Al-7Nb alloys, and comparable to that of wrought and heat-treated Ti-5Al-2.5Fe and Ti-6Al-4V alloys. The higher strength is related to the chemistry of the alloy and the actual microstructure formed as Fe is a stronger beta stabilizer than both V and Nb. Moreover, the forged and heat treated Ti-5Al-2.5Fe alloys of this study are expected to have higher interstitials contents, especially oxygen, with respect to the wrought alloy where the maximum oxygen content is commonly restricted to 0.2 wt.% [7]. Interstitials elements in Ti alloys are renowned for strengthening and embrittling the alloy by hindering dislocation movement and reducing work-hardening ability [42–44].

In terms of ductility, the strain of the forged and heat-treated Ti-5Al-2.5Fe alloy of this study is, generally, comparable to that of cast or sintered Ti-5Al-2.5Fe and Ti-6Al-7Nb alloys, and lower than that of wrought and heat treated Ti-5Al-2.5Fe alloys and Ti-6Al-4V or hot isostatically pressed Ti-5Al-2.5Fe alloy. The lower ductility is once again related to the amount of interstitials elements dissolved but also

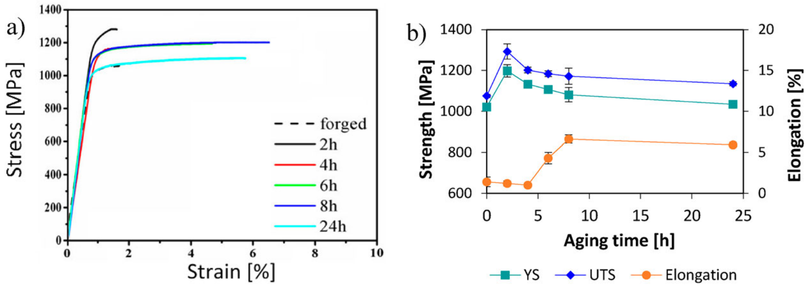

Figure 6. Mechanical properties of the Ti-5Al-2.5Fe alloy forged at 950°C: (a) representative stress-strain curves, and (b) average mechanical properties. Note: the aging time of 0 h correspond to the forged alloy.

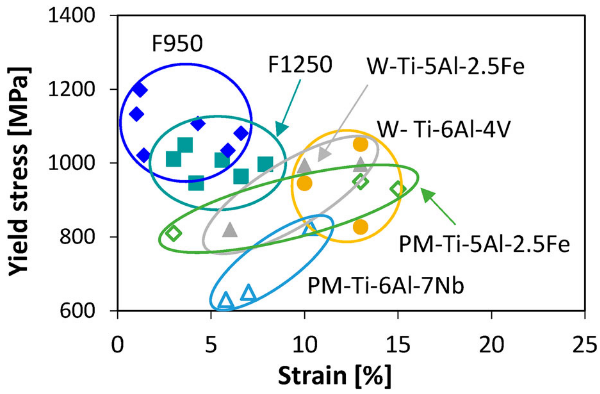

Figure 7. Comparison of the yield stress/strain values of the forged Ti-5Al-2.5Fe alloy without and with post-forging $\alpha+\beta$ solution and aging treatment with relevant α+β Ti alloys [7,24,40,41]. F – forged without and with heat treatment, W – wrought alloys including as-cast, annealed and heat treated, and PM – powder metallurgy comprising sintering and hot isostatic pressing.

further reduced by the presence of residual porosity, which is more relevant for the Ti-5Al-2.5Fe alloy forged at 950°C. Furthermore, for alloys with similar level of interstitials and residual porosity level (i.e. powder metallurgy Ti-5Al-2.5Fe and Ti-6Al-7Nb alloys), the chemistry of the alloy determines the achieved mechanical behavior due to its effect on the stabilization of the phases and the modification of the microstructural features.

Conclusions

This study investigated the production of the Ti-5Al-2.5Fe alloy, as representative of $\alpha+\beta$ Ti alloys, by means of the blended elemental approach entailing cold uniaxial pressing and sintering followed by hot thermomechanical processing by means of hot forging without and with subsequent $\alpha+\beta$ solution and aging treatments. The analysis of the microstructural evolution and of the mechanical behavior shows that the location of the forging temperature with respect to the β transus of the alloy is the most crucial parameter when manufacturing $\alpha+\beta$ Ti alloys. Forging temperatures above the β transus permit to plastically deform more the alloy, which results in materials with higher ductility but lower strength in comparison to alloys plastically deform below the β transus, specifically in the two-phase $\alpha+\beta$ region. The forging temperature or thermal history of the alloy also dictates the response of the alloy to the post-forging heat treatments, where the same trend of higher ductility but lower strength in alloys plastically deformed above the β transus is maintained. Regardless of the forging conditions, the subsequent $\alpha+\beta$ solution and aging treatment increases both the strength and the ductility reaching mechanical performance comparable to those of the equivalent wrought alloy.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability statement

All metadata pertaining to this work will be made available on request.

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