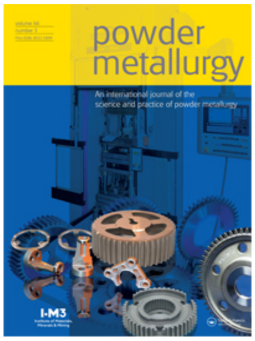

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RESEARCH ARTICLE

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Influence of powder forging and heat treatment conditions on the properties of the cost-effective Ti-5Al-2.5Fe alloy

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ABSTRACT
The Ti-5Al-2.5Fe alloy is a cheaper α+β Ti alloy with mechanical performance comparable to those of the Ti-6Al-4V alloy whose cost could be further reduced by producing it via powder metallurgy. In this study, the effect of the thermomechanical deformation temperature on the properties of the Ti-5Al-2.5Fe alloy produced from elemental powders was studied. Furthermore, the effect of the modification of the microstructure via heat treatments on the properties of the forged billets was analysed. This study demonstrates that powder forging can successfully be used to manufacture α+β Ti alloys and the selection of the forging temperature significantly affects the mechanical behaviour, where the lower the forging temperature the stronger and the less ductile the material. The post-processing via solution treatment plus aging generally improves the mechanical properties of the Ti-5Al-2.5Fe alloy, especially in terms of ductility, without compromising the strength.

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1. Introduction

Titanium (Ti) alloys have been used for a wide range of engineering applications, due to their superior combination of properties compared to other structural metals [1,2], including in the aerospace [3], automobile [4], chemical [5] and biomedical [6] industries. Regarding the latter, the Ti-6Al-4V alloy (compositions are in wt.% unless otherwise specified) has been widely employed for structural components due to its adjustable microstructure and tailorable mechanical properties by means of simple heat treatments. For example, Venkatesh et al. [7] investigated the effect of solution plus aging treatments on the Ti-6Al-4V alloy demonstrating that the strength increases and the ductility decreases if a bimodal microstructure consisting of equiaxed α grains and α+β colonies is achieved. It is worth noticing that the wide range of mechanical properties achievable in the Ti-6Al-4V alloy is due to the fact that it is a α+β alloy, which are the ones characterised by the best compromise between strength and toughness.

Although applied in industry, the high manufacturing costs of Ti alloys are hindering their wider adoption. Powder metallurgy is regarded as a cost-effective way to produce Ti alloys due to its low cost and high material utilisation. A variety of powder metallurgy methods is available to manufacture Ti alloys. For example, Henriques et al. [8] synthesised the Ti-13Nb-13Zr alloy by powder metallurgy using hydride powders. Through vacuum sintering, Henriques et al. [8] obtained relative density, tensile strength and elongation values of 97.2%, 750 MPa, 10.4%, respectively. Taddei et al. [9] produced implants by the blended elemental approach with vacuum sintering in the 900–1700°C temperature range, achieving homogeneous microstructures and good mechanical properties. Alshammari et al. [10] used cold pressing plus vacuum sintering plus forging to develop low-cost Ti-Cu alloys showing that the technique is suitable to manufacture alloys with antibacterial capability. Wen et al. [11] used vacuum furnace sintering and spark plasma sintering to consolidate a mechanically alloyed Ti-Nb-Ag powder proving that both techniques can be used to consolidate the alloy.

The development of the second generation of biomedical Ti alloys, which included the Ti-6Al-7Nb [12] and the Ti-5Al-2.5Fe [13] alloys, focused on V-free α+β titanium alloys [14] and was sparked by the reporting of the cytotoxicity of vanadium (V) in the human body [15]. In literature, some powder metallurgy techniques have been used to investigate the production of the Ti-5Al-2.5Fe alloy. For example, Siqueira et al. [16] synthesised the Ti-5Al-2.5Fe alloy by vacuum sintering using elemental powders reporting the effect of the 700–1400°C sintering temperature on the density and microstructure. A relative density

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close to 96% of the theoretical density and homogenous microstructures were achieved using 1400°C for 2 h. Hagiwara et al. [17] analysed the production of the Ti-5Al-2.5Fe alloy by the blended elemental method and the modification of the microstructure and mechanical properties by means of hot isostatic pressing. Jia et al. [18] recently proposed the use of induction heating for the production of the Ti-5Al-2.5Fe alloy demonstrating the feasibility of the proposed approach. Yamanoglu et al. [19] used the blending elemental approach to modify the Ti-5Al-2.5Fe alloy with the addition of Cu to manufacture antibacterial alloys.

From literature it is, therefore, found that the Ti-5Al-2.5Fe alloy can be used to replace the Ti-6Al-4V alloy in biomedical applications, the use of powder metallurgy is encouraged to reduce the manufacturing costs, and some studies analysed the manufacturing of the Ti-5Al-2.5Fe alloy using powder metallurgy. However, no studies investigated the thermomechanical hot deformation and the effect of subsequent heat treatments to modify the mechanical behaviour of the Ti-5Al-2.5Fe alloy produced by powder metallurgy. Consequently, the aim of this work is to prove that powder forging can be used to successfully manufacture α+β Ti alloys, as represented by the Ti-5Al-2.5Fe alloy, with satisfactorily performance. Moreover, further modification and optimisation of the microstructure and the resulting mechanical properties were investigated using β solution plus aging heat treatments.

2. Experimental procedure

In this study, the blended elemental approach was applied to produce the Ti-5Al-2.5Fe alloy with nominal composition. The raw powders used were: commercially pure Ti (O = 0.27%, N = 0.01% and H = 0.00019) with particle size <75 µm produced by means of the hydrogenation-dehydrogenation process; pure Al powder (O = 0.56%, N < 0.01% and H = 0.00003) with particle size <45 µm; and pure Fe powder (O = 0.32%, N < 0.01% and H = 0.00022) with particle size <10 µm.

The powder blends were mixed for 24 h using a two-roll mill to achieve homogeneous mixtures, which were compacted into 56 mm diameter billets at room temperature using a uniaxial pressure of 400 MPa. The pressed billets were vacuum sintered at 1250°C for 2 h under high vacuum ($\sim 10^{-4}$ Pa). The sintered billets were forged into discs at two temperatures (i.e. 950°C and 1250°C) in air. These two temperatures are, respectively, located at the upper bound of the α+β field and above the β transus of the Ti-5Al-2.5Fe alloy, which has been reported to be between 950°C [13] and 960°C [20]. The post-forging β solution plus aging heat treatment procedure included solution at 1000°C for 10 min, water quenching, and aging at 700°C. The aging times investigated were 2, 4, 6, 8 and 24 h.

X-ray diffraction (XRD) was done by means of a Philips X'Pert diffractometer to determine the constituent phases of the forged parts using a CuK_{α} radiation source and a scanning step of 0.013°. Metallographic samples were prepared by grinding and polishing. A Kroll's reagent composed of 2 ml HF, 6 ml HNO_3 and 92 ml distilled water was used to etch the samples. The microstructure was examined by optical metallography (Olympus BX60). At least three flat dog-bone-shaped tensile test specimens with cross-sectional dimensions of $2 \times 2\ mm^2$ were cut from Ti-5Al-2.5Fe billets by wire electrical discharge machining. An Instron 33R4204 universal testing machine was used to conduct the tensile testing at room temperature. A strain rate of $1 \times 10^{-4}\ s^{-1}$ was applied. During testing, the strain was measured using an extensometer with a gauge length of 10 mm.

3. Results

3.1. Microstructural evolution

Figure 1 shows the XRD patterns of the Ti-5Al-2.5Fe billets, respectively, forged at 950°C and 1250°C confirming that both as-forged parts are mainly composed of the α phase and a small amount of β phase, which is common for α+β Ti alloys [21]. Moreover, the intensity of the β phase peaks in the billets forged at 950°C is lower with respect to that of the billets forged at 1250°C.

The microstructure of the Ti-5Al-2.5Fe billets forged at 950°C and 1250°C are shown in Figure 2. It can be seen in Figure 2(a) that the alloy forged at 950°C has mainly a lamellar structure, although some elongated primary α grains typical of the bimodal microstructure of α+β Ti alloys are also present as the forging temperature is close to the β transus of the alloy. Moreover, residual pores with spherical shape, which are left by the sintering step [22] and not fully

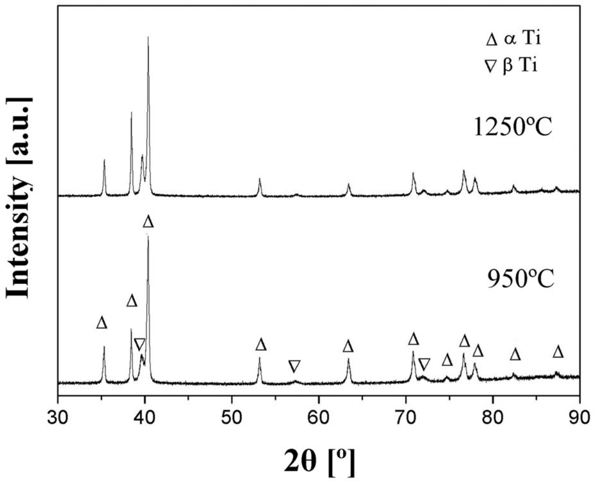

Figure 1. XRD patterns of the forged Ti-5Al-2.5Fe billets.

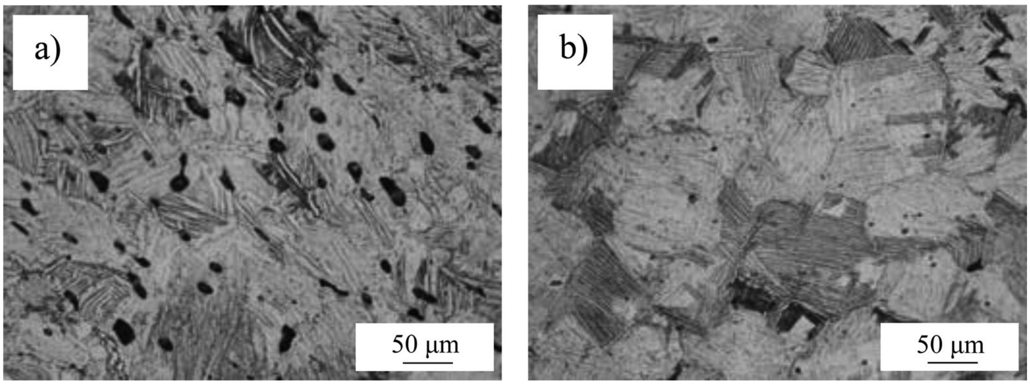

Figure 2. Microstructure of the forged Ti-5Al-2.5Fe billets: (a) 950°C and (b) 1250°C.

close by forging, can also clearly be seen in the micrograph. Concerning the Ti-5Al-2.5Fe billets forged at 1250°C (Figure 2(b)), the microstructure is fully lamellar, the size of the α grains is coarser and that of the α+β lamellae significantly refined with respect to those found in the billets forged at 950°C, and the amount of residual porosity is negligible.

The results of the microstructural characterisation performed on the Ti-5Al-2.5Fe billets forged at 950°C subjected to β solution plus aging heat treatments are shown in Figure 3 where it can be seen that the microstructure is composed of α grain boundaries and α+β lamellae [23]. The phases composing the aged Ti-5Al-2.5Fe billets are, therefore, not remarkably different from those of the forged alloy (Figure 2(a)), but their characteristics are. The increment of the aging time leads to the general coarsening of the microstructural features and, therefore, the longer the aging time the greater the width of the α grain boundaries and the coarser the α+β lamellae found within the α+β colonies. However, the overall size of the α+β colonies does not seem to be significantly affected by the aging treatment. From the micrographs of Figure 3 it can also be seen that, regardless of the aging time, the aged Ti-5Al-2.5Fe billets still have residual pores as did the forged Ti-5Al-2.5Fe billets.

From Figure 4, which shows the microstructural evolution of Ti-5Al-2.5Fe billets forged at 1250°C with the aging time, it can be seen that the material is characterised by the presence of α grain boundaries and α+β lamellae [23] as microstructural features, regardless of the actual aging time. The microstructure differs from that of the as-forged billets, but it is similar to that of the heat-treated Ti-5Al-2.5Fe billets forged at 950°C (Figure 3), at least in terms of phases. However, both the width of the α grain boundaries and the size of the α+β lamellae are finer, and the size of the α+β colonies is bigger, with respect to the heat-treated Ti-5Al-2.5Fe billets forged at 950°C. As for the latter, the increment of the aging time induces the coarsening of the microstructural features where both the width of the α grain boundaries and the size of the α+β lamellae are more affected than the size of the α+β colonies. The micrographs of the microstructural evolution show that there is a much more pronounced coarsening of the α grain boundaries at the aging time of 24 h (Figure 4(e)) with respect to the other aging times analysed, with the microstructure having a much more distinctive bimodal appearance. It is worth mentioning that, from Figure 4(d), it seems that the amount of porosity of the billets aged for 8 h is higher with respect to other aging time. This is in reality an artefact derived from the location where the sample for microstructural characterisation was actual obtained.

3.2. Mechanical properties

Typical tensile engineering stress–strain curves of the Ti-5Al-2.5Fe billets forged at 950°C and 1250°C are shown in Figure 5 where it can be seen that the billets forged at 950°C have higher strength but lower ductility in comparison to those forged at 1250°C [24]. Regardless of the forging temperature and the conditions used in the subsequent heat treatment, the Ti-5Al-2.5Fe billets are characterised by an elastoplastic behaviour. In general, the solution plus aging heat treatment improves both the strength and the ductility of the forged billets [25]; however, the specific improvement depends on both the forging temperature and the heat treatment conditions.

From Figure 6, which shows the average mechanical properties of the forged and heat-treated Ti-5Al-2.5Fe alloy, the billets forged at 950°C have yield stress (YS), ultimate tensile strength (UTS) and elongation (El) of 1021 ± 12 MPa, 1075 ± 8 MPa and 1.4 ± 0.6%, respectively. The Ti-5Al-2.5Fe billets forged at 1250°C have YS, UTS and El of 946 ± 13 MPa, 1020 ± 7 MPa and 4.2 ± 0.7%, respectively. The tensile strength of the Ti-5Al-2.5Fe billets forged at 950°C is increased significantly by the β solution plus aging heat treatment where the highest UTS of 1200 ± 11 MPa is obtained at the aging time of 8 h, with YS and El of 1144 ± 17 MPa and El of 5.7 ± 2.3%. The lowest UTS is 1155 ± 28 MPa at the aging time of 2 h, with YS and El values of 1110 ± 17 MPa and 2.3

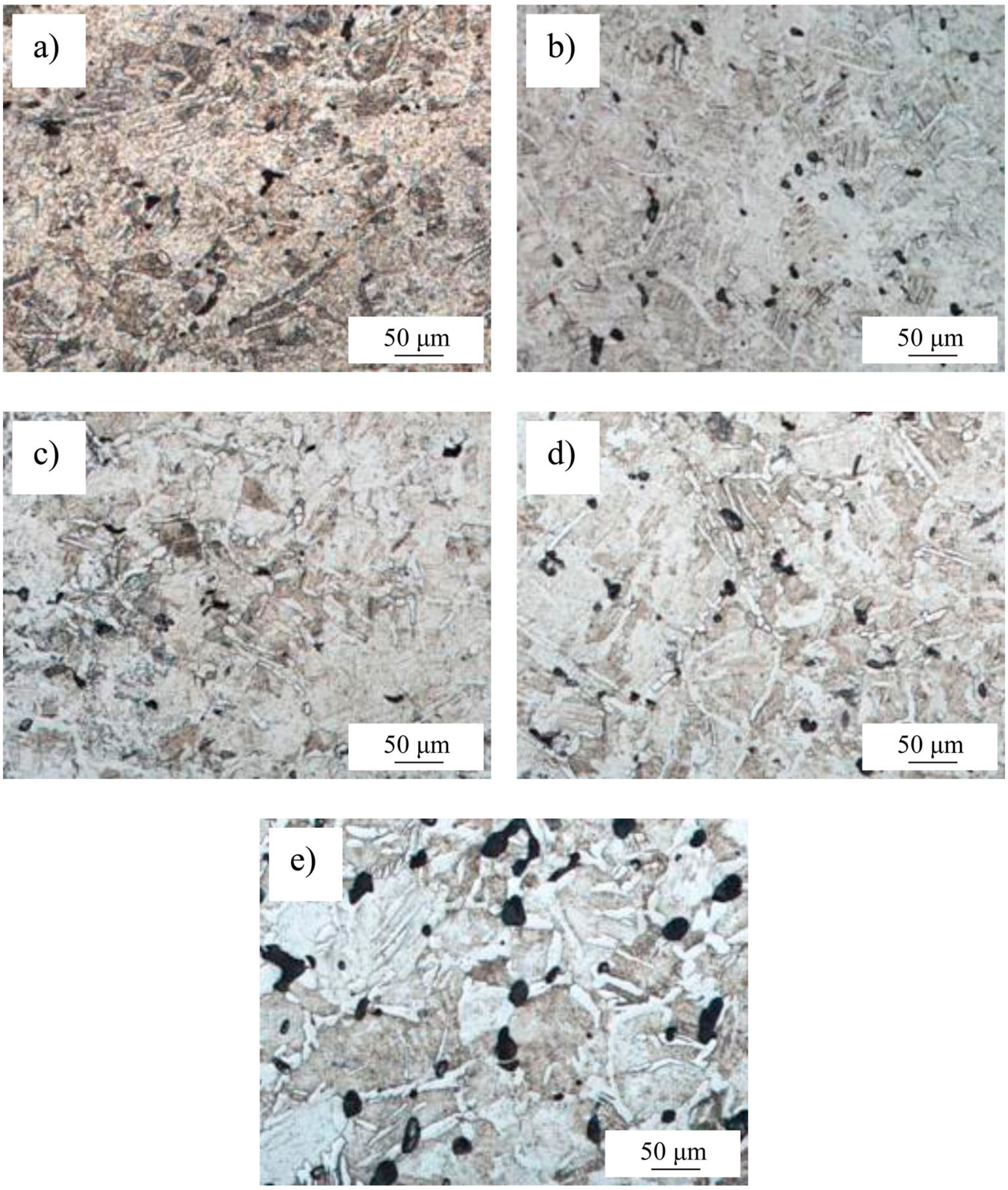

Figure 3. Microstructure of the Ti-5Al-2.5Fe billets forged at 950°C subjected to β solution plus aging heat treatment with different aging time: (a) 2 h, (b) 4 h, (c) 6 h, (d) 8 h and (e) 24 h.

± 1.0%, respectively. From Figure 6, the tensile strength of the Ti-5Al-2.5Fe billets forged at 1250°C is slightly less significantly changed by the β solution plus aging heat treatment with respect to the billets forged at 950°C. For this set of materials, the highest UTS is 1083 ± 22 MPa at the aging time of 8 h, with YS and El of 1032 ± 11 MPa and 6.5 ± 1.2%. The lowest average mechanical properties are obtained at the aging time of 2 h with YS, UTS and El values of 985 ± 6 MPa, 1043 ± 16 MPa and 7.2 ± 2.4%, respectively.

4. Discussion

4.1. Effect of the forging temperature

Forging of the sintered Ti-5Al-2.5Fe billets has two primary effects on the microstructure of the materials, it changes the phases composing the material and their ratio, as it can be seen from the XRD patterns for Figure 1, and it reduces the amount of residual porosity typical of blended elemental Ti alloys [26]. These effects are highly dependent on the selected forging temperature as it does determine the phases present during the application of the plastic deformation and, consequently, the overall deformability of the alloy. Specifically, forging at 950°C changes to slow cooled coarse lamellar structure of the sintered billets into a finer lamellar structure with a minority of elongated primary α grains still present (Figure 2(a)). The presence of the latter is because 950°C is at the upper limit of the α+β field meaning that the majority of the material already transformed into β grains. The finer lamellar structure with respect to the sintered billets is due to the faster cooling rate of the forging process. Moreover, forging at 950°C roughly reduces

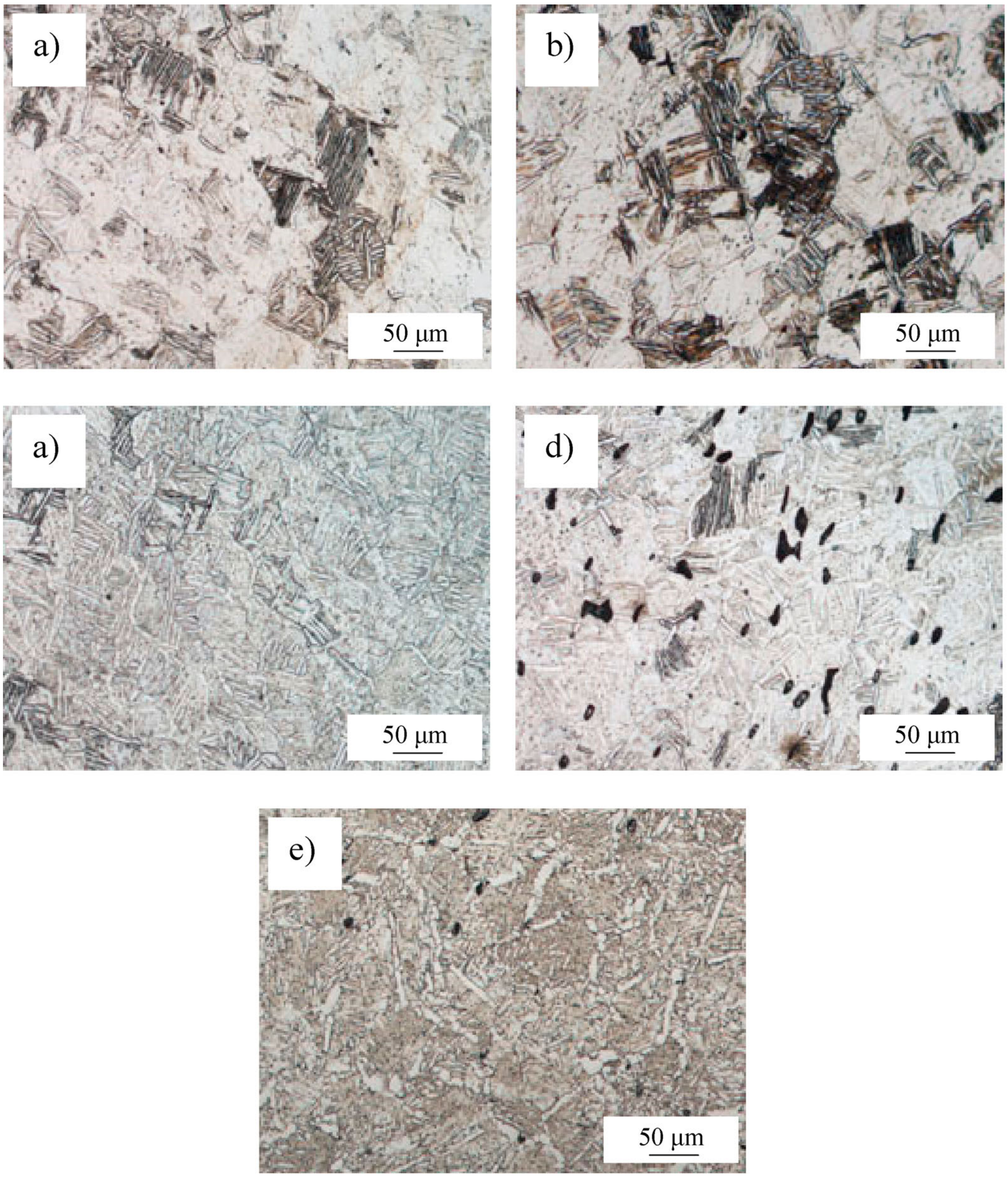

Figure 4. Microstructure of the Ti-5Al-2.5Fe billets forged at 1250°C subjected to β solution plus aging heat treatment with different aging time: (a) 2 h, (b) 4 h, (c) 6 h, (d) 8 h and (e) 24 h.

the residual porosity by half, from 8% of the sintered billets to approximately 4% of the forged billets. Forging at 1250°C, which is well above the β transus, leads to the formation of a highly refined lamellar microstructure (Figure 2(b)) in comparison to the sintered billets, due to the faster cooling rate, and a significantly lower amount of residual porosity. Furthermore, the billets forged at 1250°C also have finer lamellae but coarse α+β colonies as well as lower amount of porosity with respect to the billets forged at 950°C. This is because a higher forging temperature induces a more significant coarsening of the β grains prior to forging, it provides a much higher driving force for the nucleation of the α+β lamellae upon cooling, and it allows plastically deforming the alloy while within the β field. The latter translates into the ability to undergo more severe plastic deformation leading to the sealing of the great majority of the residual porosity left by the sintering process.

The compromise between coarser α+β colonies with finer α+β lamellae, presence of primary α grains, and a higher amount of isolated spherical residual porosity in the alloy forged at 950°C compared to that forged at 1250°C makes the former stronger but less ductile as shown by the stress–strain curves of Figure 5(a). In particular, the mechanical properties of Figure 6 show that, on average, the billets forged at 950°C have 75 MPa higher YS, 55 MPa higher UTS and 2.8% lower El in comparison to the Ti-5Al-2.5Fe billets forged at 1250°C. These results are coherent with the microstructural analysis as the pores typical of powder metallurgy materials significantly affect to ductility [27], whilst the strength of α+β Ti alloys is dependent on the colony size and on the type of phases present [28].

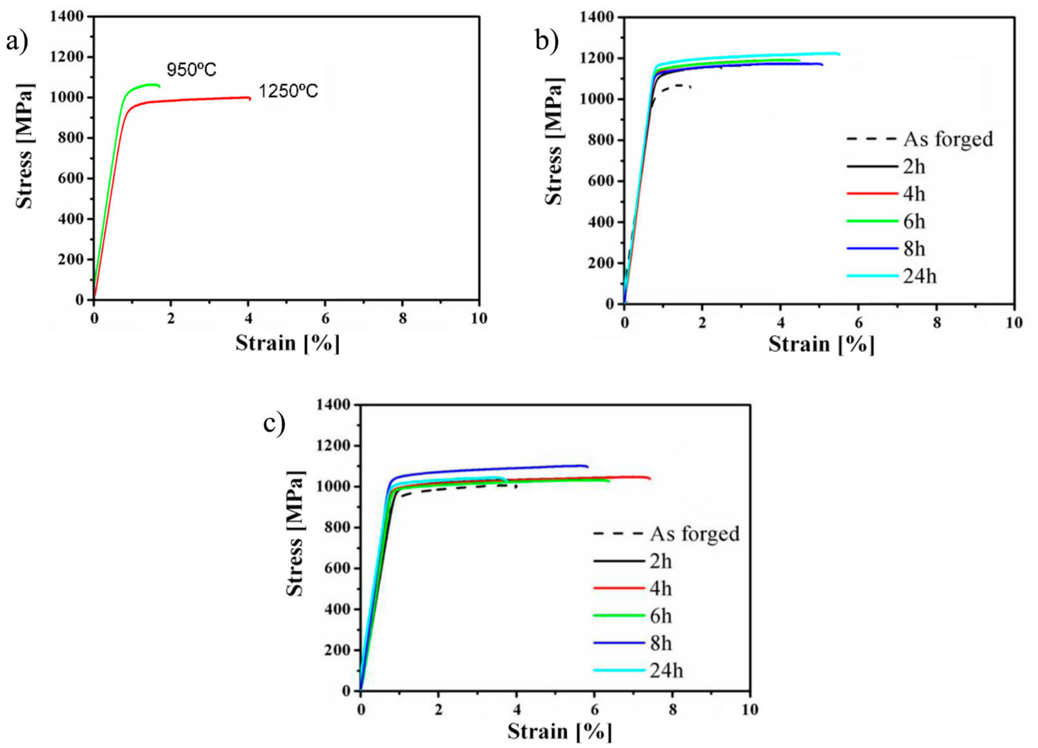

Figure 5. Representative stress–strain curves of the forged Ti-5Al-2.5Fe billets without and with β solution and aging heat treatment: (a) as-forged, (b) forged at 950°C and heat-treated and (c) forged at 1250°C and heat-treated.

4.2. Effect of the aging time

The principal effect of the post-processing heat treatment of the forged Ti-5Al-2.5Fe billets by means of β solution plus aging is the modification of the microstructure, whereas the residual porosity is not significantly affected (Figures 3 and 4). The actual modification of the microstructure is still highly influenced by the forging conditions as it determines the starting structure of the solution heat treatment. Although both Ti-5Al-2.5Fe billets forged at 950°C and at 1250°C undergo the martensitic transformation upon quenching from the β field, the size of the β

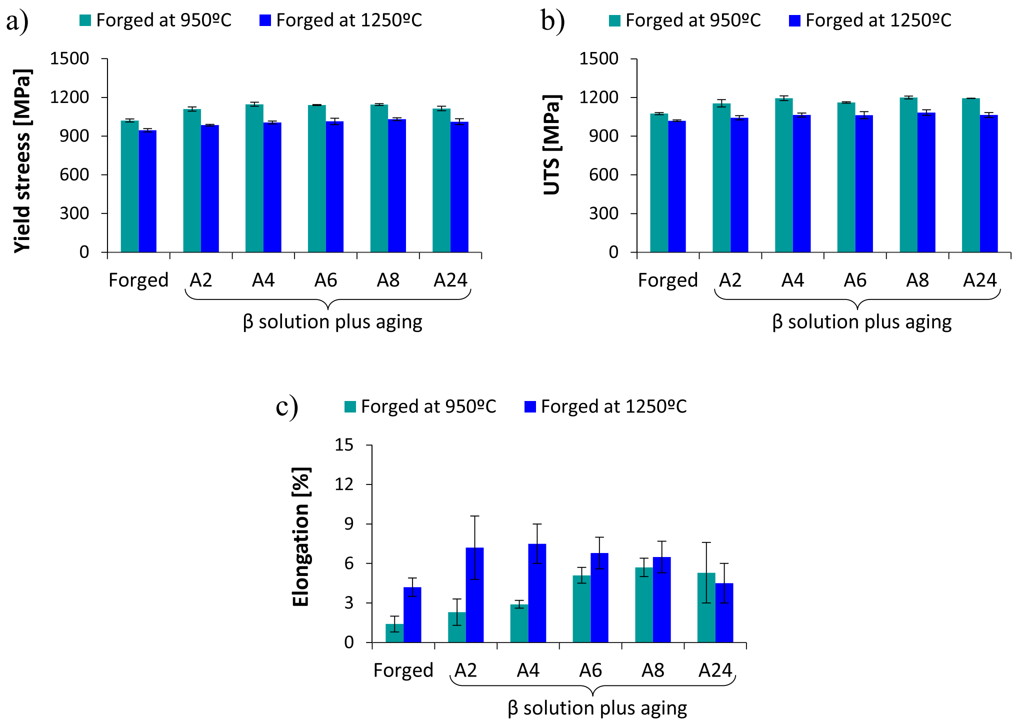

Figure 6. Average mechanical properties of the forged Ti-5Al-2.5Fe billets without and with β solution plus aging heat treatment: (a) yield stress, (b) ultimate tensile strength and (c) elongation.

grains (i.e. α+β colonies) is significantly lower in the case of the Ti-5Al-2.5Fe billets forged at 950°C as inferred from the size of the microstructure of the forged billets (Figure 2). Therefore, quenching leads to the formation of a Widmanstätten microstructure with different grain size for each alloy, even though a small amount of retained β phase could be present. Moreover, a small amount of primary α might also be present prior to quenching due to the short solution time and the fact that the powder-forged Ti-5Al-2.5Fe billets are expected to have higher oxygen content compared to the wrought Ti-5Al-2.5Fe alloy. Oxygen is α stabiliser and, consequently, increases the β transus of the alloy. Regardless of these aspects, both alloys experience the decomposition of martensite into the equilibrium phases upon heating during the subsequent aging heat treatment. Coherently, regardless of the forging conditions, after aging the microstructure of the Ti-5Al-2.5Fe billets is composed of α grain boundaries and α+β lamellae [23]; however, these microstructural features are finer, and the size of the α+β colonies bigger, in the Ti-5Al-2.5Fe billets forged at 1250°C. This is because the aging temperature of 700°C is within the α+β field. An overall progressive coarsening of the α grain boundaries and of the α+β lamellae is then found in both alloys for longer aging times, although the size of the α+β colonies is not significantly affected. A greater amount of α grain boundaries is clearly visible after 24 h of aging in both the Ti-5Al-2.5Fe billets forged at 950°C (Figure 3(e)) and at 1250°C (Figure 4(e)).

The microstructural changes described are coherent with the variation of the mechanical properties of the forged Ti-5Al-2.5Fe billets subjected to β solution plus aging. In particular, the initial decomposition of the martensitic structure to form a microstructure composed of α grain boundaries and α+β lamellae induces an overall increase of the mechanical properties in comparison to the respective forged alloy (Figure 6). This increment is more significant in terms of strength for the Ti-5Al-2.5Fe billets forged at 950°C due to the smaller size of the α+β colonies. This is the primary factor determining the strength of α+β Ti alloy, although the size of the α grain boundaries also influences the strength of α+β titanium alloy, but to a much lesser extent [28]. Consequently, aging leads to a more remarked gain in ductility in the Ti-5Al-2.5Fe billets forged at 1250°C. Both alloys experience an increase in strength for longer aging times and, once aging, the increase is slightly more significant for the Ti-5Al-2.5Fe billets forged at 950°C (Figure 6(b)). The ductility is also improved by the aging heat treatment as it continuously increases for the Ti-5Al-2.5Fe billets forged at 950°C and it plateaus after 4 h for the Ti-5Al-2.5Fe billets forged at 1250°C (Figure 6(c)). A minor reduction of the strength/strain pairs is then found for the aging time of 24 h with respect to the alloy aged at 8 h due to the coarsening of the α grain boundaries. Even if the effect of the residual porosity is not clearly detectable on the strength of the billets, the lower elongation values of the Ti-5Al-2.5Fe billets forged at 950°C with respect to the Ti-5Al-2.5Fe billets forged at 1250°C is also due to the presence of residual pores, which act as stress concentration sites [29,30].

4.3. Comparison of the mechanical behaviour

Figure 7 shows the mechanical properties (i.e. UTS vs. stain) of the forged Ti-5Al-2.5Fe billets without and with β solution plus aging heat treatment in comparison to literature. Specifically, the properties are compared to those of the wrought Ti-5Al-2.5Fe alloy under different manufacturing condition including as-cast, annealed, and β solution plus aging heat treated (β-STA) as well as those of the wrought Ti-6Al-4V alloy [31]. The as-forged Ti-5Al-2.5Fe billets have strength comparable to that of other α+β Ti alloys but the ductility is lower due to both the higher amount of interstitial elements dissolved into the powder forged Ti-5Al-2.5Fe billets as well as the residual porosity. In particular, as per the experimental

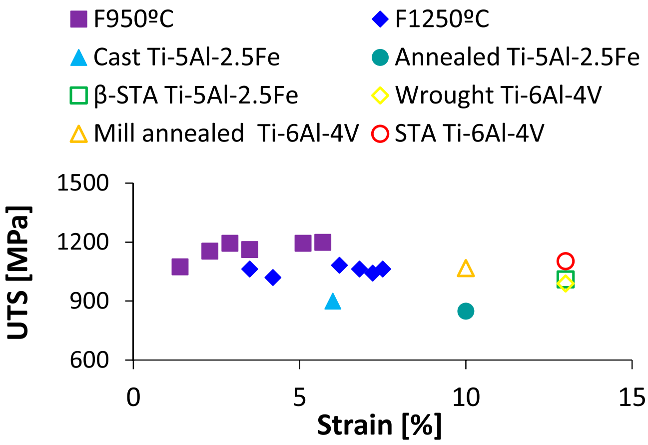

Figure 7. Mechanical properties of the forged and aged Ti-5Al-2.5Fe billets compared with literature.

procedure, the commercially pure Ti powder used has an oxygen content of 0.27 wt.%, which is already higher than 0.20 wt.%, the maximum oxygen content generally specified for α±β alloy [3]. However, the data of Figure 7 show that through the modification of the microstructural features achieved by means of the β solution plus aging heat treatment, the forged and aged Ti-5Al-2.5Fe billets reach UTS/strain pairs much closer to those of either cast or wrought α+β Ti alloys without and with different sorts of heat treatments.

5. Conclusions

This study analysed the effect that the thermomechanical deformation by α+β and β hot forging and the subsequent β solution plus aging heat treatment have on the microstructure and mechanical properties of Ti-5Al-2.5Fe billets produced through the powder metallurgy blended elemental approach. From this study, the following conclusions can, therefore, be drawn:

- The level of consolidation of the forged Ti-5Al-2.5Fe billets is improved by increasing the forging temperature, as the size and number of pores are reduced by increasing the forging temperature from 900°C to 1250°C due to higher deformability of the material at high temperatures. However, a higher forging temperature also leads to grain growth and, therefore, to the coarsening of the size of the α+β colonies, which results in the forged billets having lower strength but higher ductility.
- The β solution plus aging heat treatment changes the features of the phases and the phases composing the alloy, which are dictated by the thermal history of the alloy. In particular, the β solution plus aging heat treatment induces the coarsening of the microstructural features, including α grain boundaries and α+β lamellae, leading to general increase of the mechanical behaviour of the material regardless of the selected forging temperature. A higher increment of the mechanical properties is achieved if the α+β Ti alloy is forged at lower temperatures.

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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