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Yield of Annealing on the Properties of the Ti-5Al-2.5Fe Alloy Produced by Powder Forging

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Abstract: The high cost of titanium alloys can be reduced using alternative manufacturing techniques and using cheap alloying elements. In this study, the Ti-5Al-2.5Fe alloy, a cheaper α + β alloy than the workhorse Ti-6Al-4V, was produced by powder metallurgy combined with hot thermomechanical deformation by means of forging. The forged alloy was subsequently subjected to a heat treatment at 750 °C for several annealing times in order to modify the microstructure and tailor the mechanical properties. This study demonstrates that, regardless of the forging temperature used, annealing of the forged Ti-5Al-2.5Fe alloy improves both the strength and the ductility. Generally, the longer the annealing time, the higher the gain in strength and ductility with respect to the forged alloy. Moreover, annealing is significantly more beneficial to improve the ductility rather than the strength of the powder-forged Ti-5Al-2.5Fe alloy.

Keywords: titanium alloys; powder metallurgy; thermomechanical processing; heat treatment; mechanical properties

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1. Introduction

Titanium alloys are the material of choice for a wide range of engineering applications because they provide good combinations of properties [1,2] with respect to other metallic materials [3,4]. Specifically, Ti alloys are characterized by low density, high mechanical strength, good corrosion resistance, and biocompatibility [5,6]. However, Ti alloys are also more expensive than other structural metals and, therefore, they are commonly confined to high demanding applications, especially the ones related to the aerospace/aeronautical [7], petrochemical [8], and biomedical [9] sectors. Using cheap alloying elements [10] and alternative processing methods, such as powder metallurgy [11], are two common strategies that can be used individually, or combined, to reduce the cost of Ti alloys [12–14].

The combination of properties provided by each specific Ti alloy is dependent on the chemical composition, the thermal history of the alloy, the phases present in the material, and the manufacturing method used. Alpha Ti alloys possess better corrosion resistance, beta Ti alloys present the highest deformability, and α + β Ti alloys have the best balance between strength and toughness [15,16]. Among the different available α + β Ti alloys, the Ti-5Al-2.5Fe alloy is a direct competitor of the workhorse Ti-6Al-4V, as they are characterized by comparable mechanical properties [17]. Their yield and ultimate tensile stress are around 830 MPa and 900 MPa, respectively, and their elongation to fracture is approximately 10% in the annealed state. The Ti-5Al-2.5Fe alloy is also characterized by excellent corrosion resistance, biocompatibility, non-toxicity, and good fatigue strength. However, the Ti-5Al-2.5Fe alloy is intrinsically cheaper due to the replacement of V with Fe, as this alloy was primarily developed to be used as an implant material, especially for artificial hip and knee joints, spinal implants, and bone screws and plates, where forging is the main metallurgical production method.

As an $\alpha + \beta$ Ti alloy, Ti-5Al-2.5Fe can be heat treated in the same way as Ti-6Al-4V to alter the balance of its mechanical properties by changing the relative amount of the α and β Ti phases present, as well as their features, such as size and aspect ratio [12]. For example, by studying the effect of annealing temperatures above and below the β transus temperature and the effect of the cooling rate on the microstructure and mechanical properties of investment-cast Ti-6Al-4V alloy, Jovanović et al. [18] demonstrated that the hardness and tensile strength increase by increasing the annealing temperature and cooling rate. Annealing temperatures above the β transus temperature offered higher tensile strength but lower elongation in comparison with temperatures below the β transus. Moreover, water cooling produced higher tensile strength and lower elongation than both air cooling and furnace cooling.

The development and production of Ti alloys by means of powder metallurgy is also regarded as a promising way to reduce the cost of Ti alloys due to the intrinsic advantages of powder metallurgy. Some of the most interesting advantages for Ti alloys are the reduced number of processing steps needed to obtain a product; limited generation of expensive waste material, such as machining chips; and ability to work in the solid state, thus limiting the reactivity of Ti with processing tools. Manufacturing of the Ti-5Al-2.5Fe alloy using cold pressing and induction sintering was proposed by the authors of this work [19]. Siqueira et al. assessed the feasibility of producing the Ti-5Al-2.5Fe alloy using cold pressing and vacuum sintering [20,21] by studying its microstructural evolution and mechanical properties as a function of sintering temperature. Sintered Ti alloys are generally characterized by the presence of residual porosity and, therefore, post-processing can be considered to reduce the porosity and increase the mechanical properties [22]. Even though post-processing of the Ti-5Al-2.5Fe alloy via hot isostatic pressing has been studied [23], post-processing by means of a conventional hot deformation method, such as extrusion and forging, has not been reported.

The aim of this study is, thus, to analyze the manufacturing of the Ti-5Al-2.5Fe alloy by means of powder metallurgy, including hot forging as a post-processing step, and understand the effect of the final annealing treatment on the microstructure and mechanical properties.

2. Materials and Methods

A hydride–dehydride Ti powder (Goodfellow Ltd. Huntingdon, UK), an atomized Al powder (Ecka Granules GmbH, Velden, Germany), and a Fe carbonyl powder (Goodfellow Ltd., Huntingdon, UK), whose main features are reported in Table 1, were used as raw materials. After 24 h of two-roll blending, the Ti-5Al-2.5Fe powder blends were shaped at room temperature using an applied uniaxial pressure of 400 MPa. The samples were then vacuum sintered using the following conditions: 1250 °C/2 h at 10^{-4} Pa heated at 10 °C/min. The sintered Ti-5Al-2.5Fe alloy was characterized by the typical $\alpha + \beta$ lamellar microstructure [24,25] and a value of relative density of 92%, which is common for sintered Ti alloys [26,27].

Table 1. Features of the starting powders as per suppliers' specification.

Powder	Morphology	Max Particle Size	Purity
Ti	Angular	<75 μm	>99.4%
Al	Spherical	<45 μm	>99.7%
Fe	Dendritic	<10 μm	>99.7%

The sintered alloys were subsequently hot plastically deformed using a forging ratio of ~2, for which the samples were induction heated up to either 950 °C or 1250 °C. It is worth noticing that by using these two temperatures, the Ti-5Al-2.5Fe alloy was plastically deformed with either a bimodal or an equiaxed microstructure, respectively [28].

The β transus of the Ti-5Al-2.5Fe alloy has been reported to be between 950 °C [29] and 960 °C [30]; therefore, 950 °C sits at the upper bound of the two-phase α + β region, meaning that a small portion of primary α grains are still present in the alloy, which is not typical for sintered Ti alloys [9,28]. Consequently, the microstructure of the alloy forged at 950 °C was characterized by a small amount of primary α Ti grains elongated along the direction perpendicular to the applied plastic deformation load and coarse lamellar structure.

Furthermore, forging at 950 °C was partially effective in sealing the residual porosity, increasing the value of the relative density to approximately 96%. Forging of the Ti-5Al-2.5Fe alloy at 1250 °C resulted in a highly refined fully lamellar microstructure composed of coarser α grains and significantly finer α + β lamellae with respect to the alloy forged at 950 °C. This is due to the fact that the alloy spent time at high temperature, which led to a coarsening of the prior β grains and cooling down fast from high temperature, reducing the time for coarsening the α + β lamellae [31,32]. Due to much higher deformability, the Ti-5Al-2.5Fe alloy forged at 1250 °C was fully dense as the applied forging load was able to seal the residual porosity, reaching a relative density value of 99.8%.

After forging, a final annealing heat treatment at 750 °C was performed every two hours for up to 10 h in order to modify the microstructure and, therefore, analyze the variations in mechanical behavior. After annealing, the samples were furnace cooled. In order to carry out the microstructural analysis, the classical metallographic preparation method was used, and the samples were etched using a Kroll's reactant comprising 2 mL of HF and 6 mL of HNO_3 dissolved in 92 mL of distilled water. Microstructural analysis was performed on an Olympus BX 60 optical microscope (Olympus, Auckland, NZ) equipped with a digital camera. In order to quantify the mechanical behavior of the forged and annealed Ti-5Al-2.5Fe alloy, dogbone test-pieces with a rectangular cross section of 2 × 2 mm^2 and 20 mm gauge length were cut by means of electrical discharge machining. The surfaces of the tensile test-pieces were subsequently ground to eliminate any effect from the machined surfaces. Room-temperature tensile testing was performed by applying a strain rate of 1×10^{-4} s^{-1} by means of an Instron 33R4204 universal testing machine (Instron, Norwood, MA, USA). An external mechanical extensometer was used to record the elongation, and the offset method was used to quantify the yield stress. A minimum of three tensile test-pieces were tested in order to be able to calculate the average tensile properties and their variability.

3. Results

Annealing of the Ti-5Al-2.5Fe alloy forged at 950 °C leads to the formation of a fairly coarse lamellar microstructure composed of some primary α Ti grains and α + β colonies (Figure 1). The microstructure found in the annealed samples does not significantly differ from that of the forged alloy. The increment of the annealing time up to 10 h has a relatively minor effect on the microstructure evolution of the Ti-5Al-2.5Fe alloy forged at 950 °C. This results in the coarsening of the microstructural features, including the primary α Ti grains and the α + β lamellae. From Figure 1, it can also be seen that the annealed Ti-5Al-2.5Fe alloy forged at 950 °C is still characterized by the presence of residual pores, which are also mildly affected by the annealing heat treatment through pore rounding.

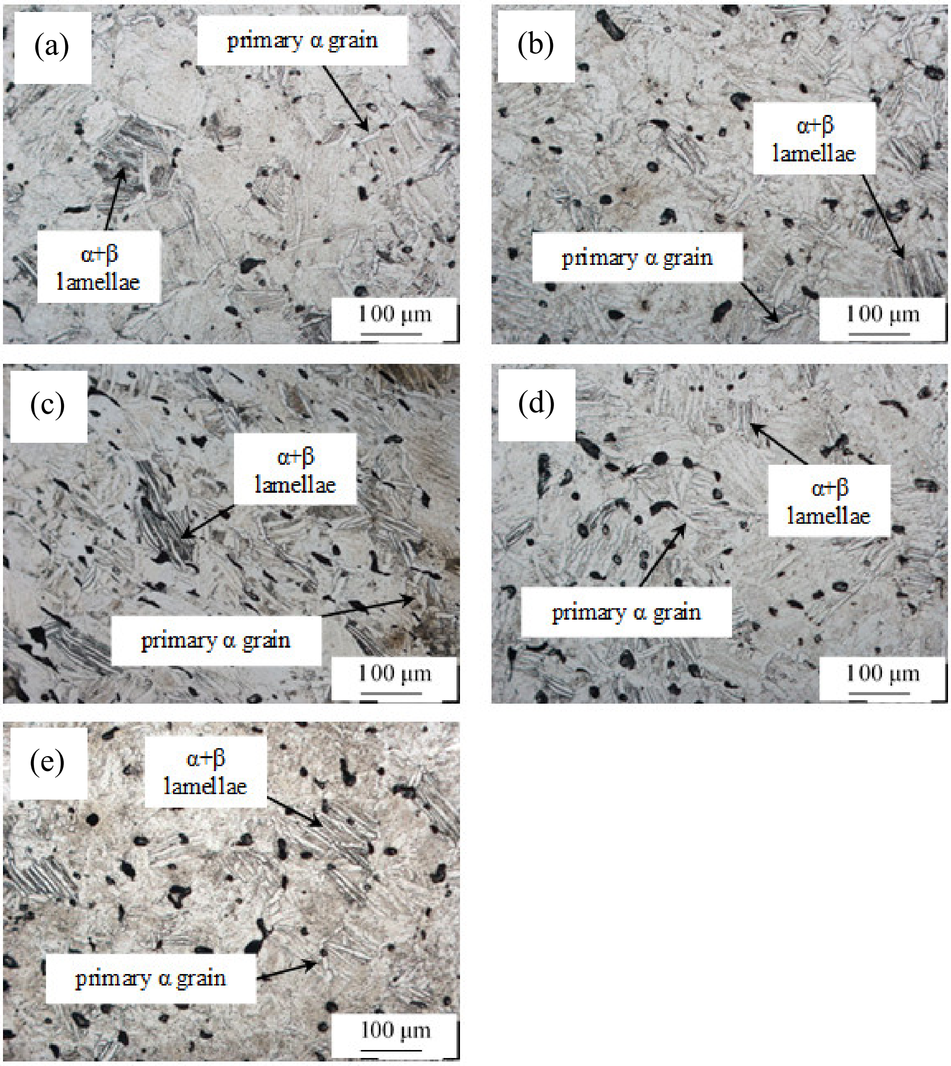


Figure 1. Microstructure of the Ti-5Al-2.5Fe billets forged at 950 °C subjected to β annealing at 750 °C with different aging times: (**a**) 2 h, (**b**) 4 h, (**c**) 6 h, (**d**) 8 h, and (**e**) 10 h.

From the analysis of the evolution of the microstructure of the Ti-5Al-2.5Fe alloy forged at 1250 °C with the annealing heat treatment time (Figure 2), it can be seen that alloy is characterized by a fine lamellar microstructure, which coarsens as the annealing time increases. It can also be seen that the annealing at 750 °C, thus within the two-phase *α* + β region, leads to the nucleation and growth of *α* Ti grain boundaries, which start to be distinguishable in the microstructure after 6 h of annealing heat treatment (Figure 2c). Although both alloys experience a coarsening of the microstructural features, the Ti-5Al-2.5Fe alloy forged at 1250 °C has a significantly finer microstructure in comparison with the Ti-5Al-2.5Fe alloy forged at 950 °C. Furthermore, coherently with the microstructure of the forged alloys, the former is fully dense, and the few small pores present are not affected by the annealing heat treatment.

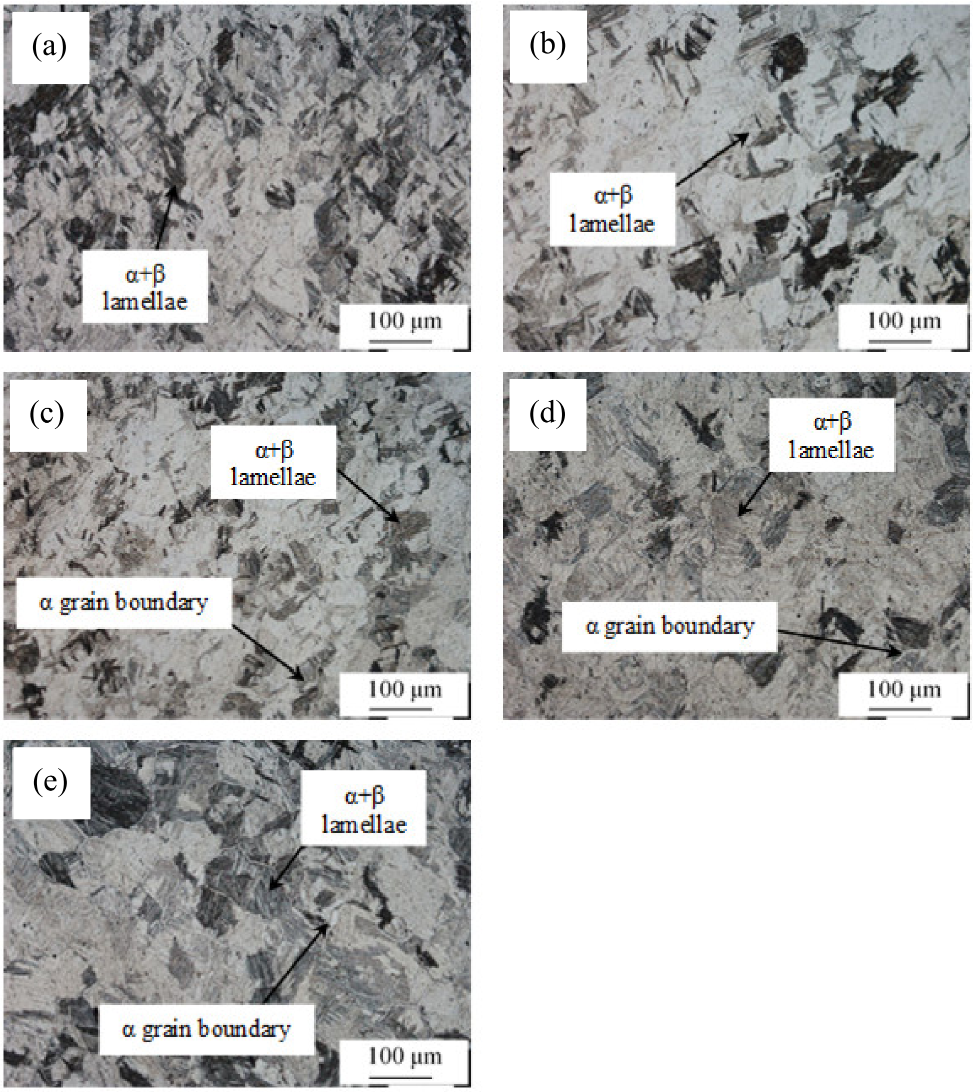


Figure 2. Microstructure of the Ti-5Al-2.5Fe billets forged at 1250 °C subjected to β annealing at 750 °C with different aging times: (**a**) 2 h, (**b**) 4 h, (**c**) 6 h, (**d**) 8 h, and (**e**) 10 h.

Figure 3 shows the representative stress–strain curves of the forged Ti-5Al-2.5Fe alloy without and with subsequent annealing heat treatment, where it can be seen that, regardless of the manufacturing conditions, the alloy is characterized by both elastic and plastic deformation. The annealing heat treatments have a higher impact on the yield stress of the Ti-5Al-2.5Fe alloy forged at 950 °C compared with that of the alloy forged at 1250 °C. The latter seems to have consistently lower strength but higher ductility in comparison with the former.

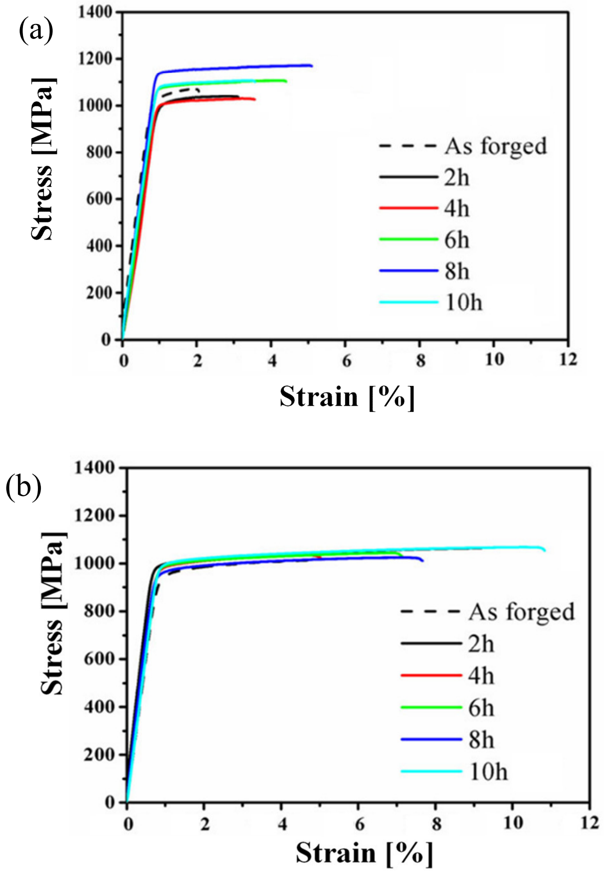
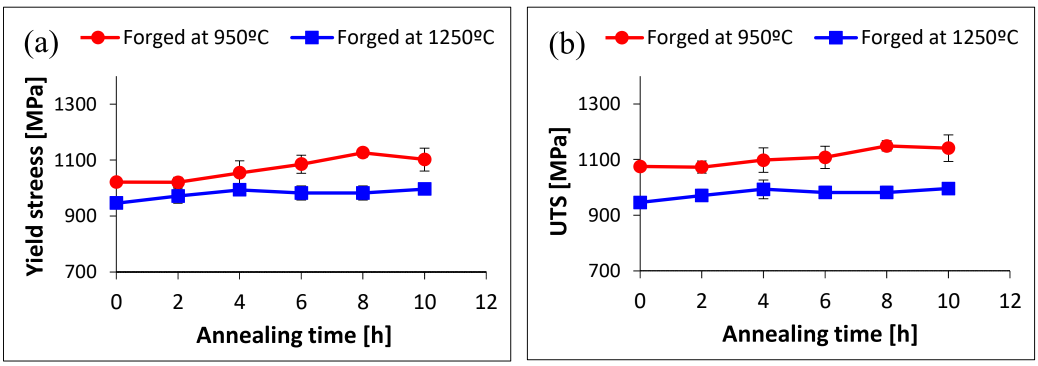


Figure 3. Representative stress–strain curves of the Ti-5Al-2.5Fe alloy forged at (**a**) 950 °C and (**b**) 1250 °C without and with annealing treatment.

The values of the average mechanical properties, including yield stress (YS), ultimate tensile strength (UTS), and elongation to fracture (El), are displayed as a function of the annealing time in Figure 4. It is worth noticing that the annealing time of 0 h refers to the Ti-5Al-2.5Fe alloy in the as-forged state. The Ti-5Al-2.5Fe alloy forged at 950 °C has YS, UTS, and El of 1021 MPa, 1075 MPa, and 1.4%, respectively. The Ti-5Al-2.5Fe alloy forged at 1250 °C has YS, UTS, and El of 946 MPa, 1020 MPa, and 4.2%, respectively. Generally, the annealing treatment improves both the strength and the ductility of the forged Ti-5Al-2.5Fe parts, regardless of the forging temperature and the annealing time. However, a higher increase in strength is found for the samples forged at 950 °C (Figure 4a), while a higher increase in ductility is found for the samples forged at 1250 °C (Figure 4c).



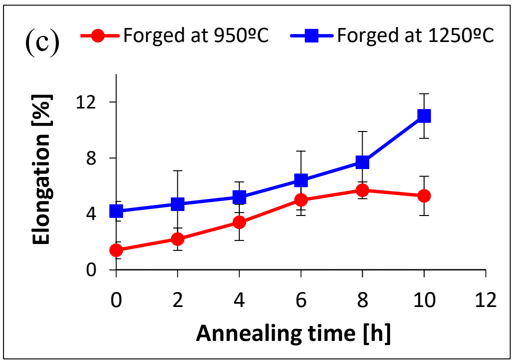


Figure 4. Variation in the mechanical properties of the forged Ti-5Al-2.5Fe alloy as a function of the annealing time: (**a**) yield stress, (**b**) ultimate tensile strength, and (**c**) elongation to fracture.

Apart from the forged samples, the lowest mechanical properties for the Ti-5Al-2.5Fe alloy forged at 950 °C are obtained after an annealing heat treatment of 2 h, reaching YS, UTS, and El values of 1020 ± 18 MPa, 1073 ± 22 MPa, and 2.2 ± 0.8%, respectively. The highest mechanical properties are achieved by means of 8 h of annealing heat treatment as YS = 1126 ± 16 MPa, UTS = 1149 ± 19 MPa, and El = 5.7 ± 0.6%. Similarly, in the case of the Ti-5Al-2.5Fe alloy forged at 1250 °C, with the exception of the forged alloy, the lowest mechanical properties of YS = 971 ± 26 MPa, UTS = 1042 ± 22 MPa, and El = 4.7 ± 2.4% are attained after annealing for 2 h. The annealing heat treatment of 10 h permits us to obtain the highest mechanical performance in the Ti-5Al-2.5Fe alloy forged at 1250 °C, which, for the YS, UTS, and El, are 996 ± 5 MPa, 1068 ± 2 MPa, and 11.0 ± 1.6%, respectively.

4. Discussion

In this study the α + β Ti-5Al-2.5Fe alloy was produced using the conventional powder metallurgy route of cold uniaxial pressing plus sintering followed by hot forging. The selection of the forging temperature significantly affects the outcome of the plastic deformation process as it determines the phases present, as well as the deformability of the material.

Specifically, hot forging of the Ti-5Al-2.5Fe alloy at a relatively low temperature, 950 °C in this study, leads to the formation of a coarse lamellar microstructure with a small amount of primary α Ti grains still present in the microstructure, which can be regarded as a bimodal microstructure, and the partial closure of the residual porosity left by the sintering process. Both aspects are related to the fact that 950 °C is located just below the β transus temperature, where the majority of the material is already transformed into β but a small portion is still α. The untransformed primary α Ti grains remain in the forged microstructure, although their spherical morphology is changed to elongated, while the rest of the material is transformed into α + β lamellae upon cooling. The coarse size of the lamellae is the compromise between a low-nucleation driving force due to the low forging temperature and fairly high cooling rate imposed by forging in air. The actual amount of the reduction in the residual porosity is the compromise between the applied forging load and relatively limited plastic deformability of the alloy at the forging temperature of 950 °C.

From Figure 1, the post-processing annealing heat treatment at 750 °C of the Ti-5Al-2.5Fe alloy forged at 950 °C does not significantly modify the phases and the porosity of the forged alloy. Specifically, the microstructure of the annealed Ti-5Al-2.5Fe alloy is still composed of primary α Ti grains and α + β colonies, as well as spherical pores. This is due to the fact that the selected annealing temperature is within the two-phase α + β region and it is only just above 50% of the absolute melting temperature of the alloy, where diffusion is still sluggish. However, the increment of the annealing time leads to some coarsening of the primary α Ti grains and α + β lamellae found within the colonies; however, it does not affect the residual porosity. Modification of the forged microstructure, which was formed under fairly fast cooling, as well as coarsening during the post-processing annealing heat treatment, results in the linear increase in the mechanical properties of the Ti-5Al-2.5Fe alloy forged at 950 °C with the annealing time. More in detail, annealing for 2 h does not improve the strength but increases the ductility, while both the strength and the ductility are enhanced for longer annealing times. The continuous improvement of 20–30 MPa in terms of YS and UTS and approx. 1% in elongation every 2 h of annealing heat treatment plateaus at 8 h, and it is due to the coarsening of the microstructural features.

Hot forging of the Ti-5Al-2.5Fe alloy in the β field, which is 1250 °C in the current study, leads to the formation of a highly refined lamellar microstructure composed of very fine α + β lamellae combined with the simultaneous complete sealing of the residual porosity. Once again, both aspects are related to the combination of high forging temperature and the deformability of the material. Forging at 1250 °C means that the alloy is purely composed of β grains while the uniaxial load is applied, the driving force for nucleation of the α + β lamellae is significantly higher, the cooling rate experienced during forging in air is also much higher, and so is the deformability of the alloy. As in the case of the Ti-5Al-2.5Fe alloy forged at 950 °C, the post-processing annealing heat treatment of the Ti-5Al-2.5Fe alloy forged at 1250 °C has only a minor impact on the phases present in the microstructure (Figure 2). In particular, annealing eliminates any potential metastable phases/grains that could have formed due to the fast cooling from the forging temperature and leads to the coarsening of the microstructural features as the annealing time increases. As the annealing heat treatment is performed within the two-phase α + β region, the widening of α Ti grain boundaries occurs along with the coarsening of the α + β lamellae. The microstructural changes induced by the annealing at 750 °C leads to the steady increase in the mechanical properties of the Ti-5Al-2.5Fe alloy forged at 1250 °C, with an average increment of 20 MPa in YS and UTS and 1.3% in elongation every 2 h of annealing heat treatment.

The comparison of the microstructure and properties of the Ti-5Al-2.5Fe alloy forged at 950 °C and 1250 °C clearly indicates that, in the current investigation, the selected forging temperature is the parameter with the highest influence on the performance of the alloy. In particular, the forging temperature determines the starting microstructure for the subsequent annealing heat treatment, which only mildly affects the microstructural features, primarily leading to their coarsening.

Consequently, the Ti-5Al-2.5Fe alloy forged at 950 °C has higher strength but lower ductility compared with the alloy forged at 1250 °C (Figure 3) because of the untransformed primary α Ti grains of the bimodal microstructure and the presence of the residual porosity [33,34]. The phases present in the forged alloys and the characteristics of these phases both affect the response of the alloy to annealing and are affected by the annealing heat treatment. Therefore, although both the strength and the elongation of the forged alloys progressively increase with the annealing time, annealing increases more the strength of the Ti-5Al-2.5Fe alloy forged at 950 °C, and increasing more the ductility of the Ti-5Al-2.5Fe alloy forged at 1250 °C.

The mechanical properties of the forged and annealed Ti-5Al-2.5Fe alloy of the current study are compared with the literature in Figure 5. Through the optimization of the heat treatment, higher strength values can be achieved in the powder-forged Ti-5Al-2.5Fe alloy compared with alloys in the literature subjected to similar heat treatment procedures. The values of the ductility of the powder-forged Ti-5Al-2.5Fe alloy are generally lower compared with those found in the literature, as the latter refer to the wrought Ti-5Al-2.5Fe alloy. Most of the strength/ductility pairs found for the forged and annealed Ti-5Al-2.5Fe alloy are comparable with those of other wrought α + β Ti alloys, including the workhorse Ti-6Al-4V alloy, making the manufacturing of α + β Ti alloys through powder forging a valuable alternative for producing Ti alloys for non-critical structural engineering applications.

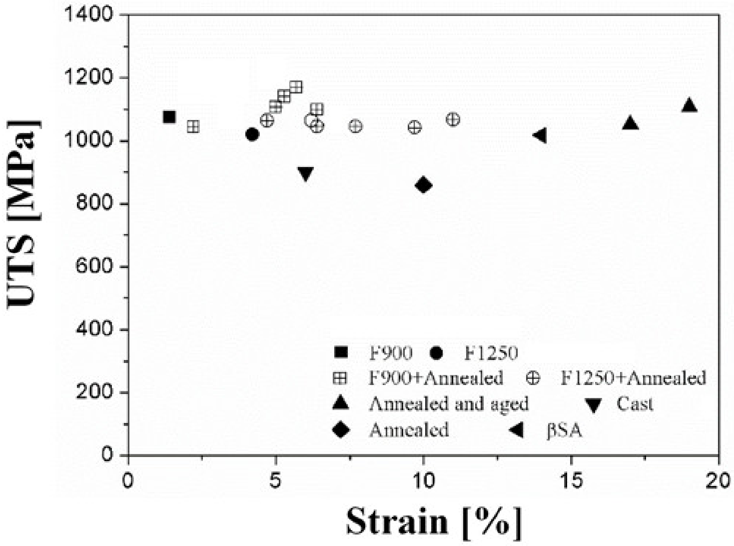


Figure 5. Comparison of the mechanical properties of the forged and annealed Ti-5Al-2.5Fe alloy, data from [12,28].

5. Conclusions

In this study, the α + β Ti-5Al-2.5Fe alloy was produced by means of the powder metallurgy blended elemental approach, including cold uniaxial pressing and sintering, thermomechanical deformation through hot forging, and a final annealing heat treatment. The analysis of the microstructure and of the mechanical properties of the forged and annealed Ti-5Al-2.5Fe alloy reveals that the selection of the hot forging temperature is crucial. Specifically, the higher the forging temperature, the more deformable to alloy, which is beneficial for reducing the residual porosity; however, this leads to a more pronounced grain growth. Consequently, the lower the forging temperature, the higher the strength and the lower the ductility of the alloy. Specifically, the tensile strength and elongation of the Ti-5Al-2.5Fe alloy forged at 950 °C and 1250 °C are 1075 MPa and 1.4%, and 1020 MPa and 4.2%, respectively.

Regardless of the chosen forging procedure, the annealing post-processing heat treatment progressively increases both the strength and the ductility with the annealing time, reaching the highest tensile strength of 1149 MPa and 1068 MPa for the Ti-5Al-2.5Fe alloy forged at 950 °C and 1250 °C, respectively. However, higher gains in strength and ductility are achieved when starting from the alloy forged at low and high temperatures, respectively. This study demonstrates that powder forging is a viable alternative process to manufacture Ti alloys with mechanical properties comparable to those of wrought α + β Ti alloys.

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Data Availability Statement: All metadata pertaining to this work will be made available on request.

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