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Influence of α-Phase Field Heat Treatment on the Tensile and Primary Creep Resistance of a Powder Metallurgical Processed Ti-45Al-5Nb-0.2B-0.2C Titanium Aluminide Alloy

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Abstract. In powder metallurgical processing the sintering process, as well as heat treatments, can drastically influence microstructure formation. In the case of γ-titanium aluminides, it is critical to achieve certain microstructure parameters, such as colony size, porosity and grain boundary morphology in order to obtain appropriate mechanical properties. In this study, the effect of a heat treatment implemented after sintering with the objective of varying the colony size was investigated. Specimens of Ti-45Al-5Nb-0.2B-0.2C prepared by metal injection moulding and uniaxial pressing of feedstock were used to evaluate the tensile and creep properties. Heat treatments conducted at 1350 and 1400 °C for 3 h led to colony sizes of approximately 100 and 200 μm, respectively. Classically, there is an inverse relationship between grain size and creep resistance, nonetheless, for γ-titanium aluminides, the morphology of the colony boundaries was also found to play a role. The larger colony sizes achieved with the heat treatments did not improve the primary creep resistance, which was explained by the change in the morphology of the colony boundaries as they became larger.

Introduction

Titanium aluminide alloys represent an emerging class of lightweight materials with an excellent balance between high specific strength, high stiffness, good oxidation resistance, resistance against ignition and good creep properties. Therefore, γ-based titanium aluminides are highly considered to replace the well-established Ni-based superalloys at moderately high temperatures of about 600-800 °C for certain applications. However, depending on the application conditions, the mechanical properties of titanium aluminides have to be tailored or improved for the temperature range above 700 °C due to microstructural instabilities, which directly affect creep properties and oxidation resistance. To achieve this, thermomechanical processing followed by heat treatment has been successfully applied leading to favourable results [1].

Furthermore, due to the inherent brittleness at room and small plasticity at higher temperature, the difficulty in shaping these materials poses a great challenge. In this context, powder metallurgy techniques, such as metal injection moulding (MIM) may help to solve some of the processing difficulties found in ingot metallurgy. MIM can deliver sound mechanical properties with good microstructural and chemical homogeneity [2–4]. Additionally, sintering and heat treatment can be conducted in the same thermal cycle and, since MIM is a near-net-shape technique, further machining is often unnecessary. Thus, by reducing processing times significant cost savings can be achieved.

In terms of creep resistance, the superiority of fully lamellar morphology is well established. A colony of such morphology consists of alternating lamellae of γ-TiAl and α_2-Ti_3Al phase. Both intermetallic phases coexist in the composition range of ~35 to 50 at% Al, from room temperature up to 1150 °C, the eutectoid temperature. By heat-treating in the single α-phase field an alloy, which lies in the aforementioned composition range, and subsequently cool down below the eutectoid temperature, such a lamellar microstructure can be adjusted [1]. The colony boundaries of such a structure often present an interlocking arrangement of lamellae of neighbouring colonies and alloys with such an interlocked lamellar microstructure are less susceptible to intergranular crack propagation and grain boundary sliding. This in turn influences directly the room temperature mechanical behaviour and creep mechanisms [1,5].

For a good combination of room temperature properties and high temperature creep resistance, lamellar spacing and colony size need to be controlled. If high temperature and/or long time heat treatments are employed in the α-phase field, excessive grain growth might occur resulting in coarsened lamellar colonies in the range of 200-1000 µm [6]. Furthermore, the lamellar spacing is directly related to the cooling rate and typically, the higher the cooling rate, the thinner are the formed lamellae [1]. It is classically established that larger grains are beneficial for creep resistance, considering that mechanisms such as Coble creep and grain boundary sliding are dependent on grain size and that grain boundaries are preferred locations of creep porosity. In fact, for all diffusion assisted processes the grain size is generally an important parameter [7]. On the other hand, Maruyama et al. [8,9] assessed the influence of the grain size (keeping the same lamellar spacing) on the creep behaviour of γ-titanium aluminides concluding that the creep rate may increase when the size is <100 µm, becoming independent for grain sizes above this value. Additionally, Prasad and Chaturvedi [6] showed that, for a Ti-45Al based alloy annealed at 1350 °C, as the annealing time increased, the colony boundaries became increasingly planar, which could influence the creep behaviour. The authors, however, did not characterise the mechanical properties. Consequently, given the novelty of MIM processed materials for high-temperature applications, an investigation concerning variations in colony sizes and boundary morphologies is important to elucidate if the reported phenomena are also valid for this alternative processing technique.

In this research work, the influence of different heat treatments on the growth and morphology of the lamellar colonies in a powder metallurgical processed titanium aluminide alloy, Ti-45Al-5Nb-0.2B-0.2C (TNB-V5), was assessed. The resulting mechanical properties (tensile strength at room temperature and primary creep) were evaluated for samples heat treated at different annealing temperatures, however, employing the same holding time and cooling rate down to the $\alpha_2 + \gamma$ phase field.

Materials and Methods

An ingot of TNB-V5 (Ti-45Al-5Nb-0.2B-0.2C, in at%) was prepared by GfE GmbH, Nürnberg, Germany, and atomised at the Helmholtz-Zentrum Geesthacht using the EIGA (electrode induction melting gas atomization) process [10]. Two kinds of samples were prepared for the tests: cylindrical (for compression creep tests) and tensile specimens. The first were uniaxially pressed and the last one injection moulded, both using the same feedstock with approximately 8 wt% of binder (27 vol%).

Chemical debinding was done at 40 °C using hexane as a solvent medium. Thermal debinding, sintering and heat treatments were carried out in the same equipment, a high-vacuum cold-wall furnace XERION XVAC, with tungsten heating elements and internal molybdenum shield packs. The thermal debinding was performed between 450 and 600 °C and sintering at 1500 °C for 2 h.

From here on, HT1 refers to the sample heat treated at 1350 °C and HT2 at 1400 °C, whereas Reference denotes the sample only sintered (without heat treatment). After sintering at 1500 °C, the Reference was cooled down to 1000 °C at 100 K/min and then to room temperature by furnace cooling. Alternatively, after sintering, HT1 and HT2 samples were cooled down to the heat treatment temperatures at a rate of 100 K/min. In both cases, a dwell time of 3 h was applied, followed by furnace cooling. For each heat treatment condition, six samples were prepared, three for tensile and three for creep tests.

The temperatures 1350 and 1400 °C lie in the α-phase field according to differential scanning calorimetry measurements. In this temperature range, colony growth is more pronounced and consequently changes of the mechanical properties are expected in the final sintered specimens. Subsequent cooling down to 1000 °C and room temperature were done in the same manner as the Reference, which assumingly led to a similar lamellar spacing.

Creep tests were conducted at 800 °C and 350 MPa. Using an Instron-Satec SF-16 2230 creep test machine. Tensile tests were performed at room temperature by means of a Schenck Trebel RM 100 tensile test machine with a load cell of 100 kN and deformation rate of 0.1 mm/min. The specimen gauge length was 30 mm with a diameter of 4.3 mm. Strain was measured by a laser extensometer Fiedler Laser Scanner.

Microstructural characterization was conducted by light optical microscopy using an Olympus PMG-3 microscope. Both porosity and colony size were measured by the AnalySIS Pro software, the former feature being estimated from micrographs taken with polarised light via the intercept method according to the ASTM 112-13 standard [11].

Results and Discussion

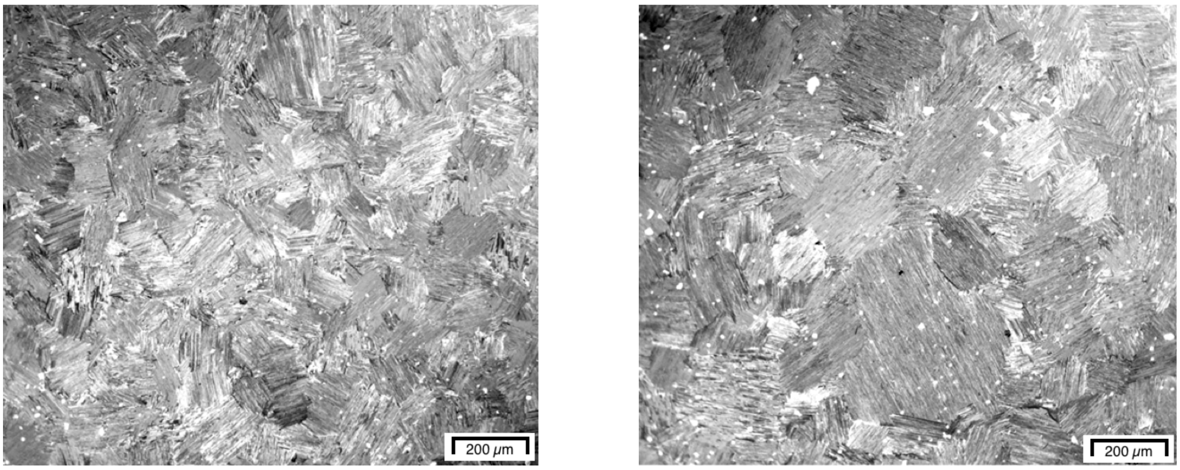
The average colony sizes obtained after sintering and heat treatments are shown in Table . Clearly, HT1 showed a slightly larger colony size than the Reference, whereas HT2 exhibits much larger colonies than both previous samples, even considering the large data scatter. Typically, long exposure times in the α-phase field result in long times available for diffusional processes to take place. Hence significant grain growth is expected to occur. Reportedly this gives rise to grains in the range size of 200-1000 µm [6] for various Ti-45Al alloys.

Table 1. Average colony size of different processing conditions.

Specimen	**Reference**	**HT1**	**HT2**
Average colony size (µm)	81.0 ± 8	105.3 ± 12	192.0 ± 45

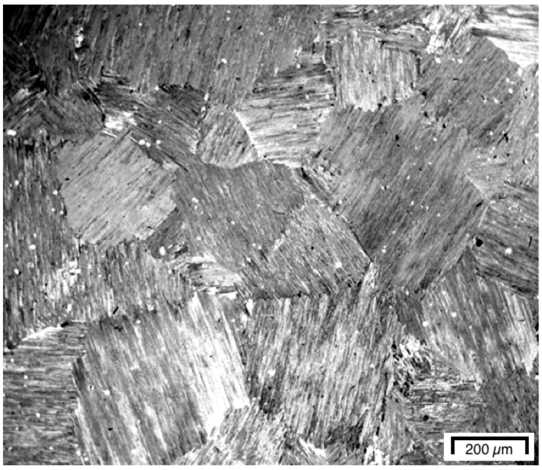
Fig. 1 shows light optical micrographs of sintered specimens with the same magnification. **Fig. 1**a corresponds to the Reference, while **Fig. 1**b and 1c show the heat-treated conditions. The measured porosity is also indicated in each case. With the different heat treatments, the colony size and boundary morphologies were remarkably distinct. Nonetheless, the porosity level was in the same range: lower than 1%.

Previous studies involving powder metallurgy processing of γ-titanium aluminides, which were performed to understand the effect of the heat treatment on the microstructure evolution, reported similar observations as displayed in **Fig. 1**. Dudzinski et al. [12] used gas atomised TiAl powder with varied compositions and consolidated by hot isostatic pressing (HIP) at 1250 °C for 2 h. Prasad and Chaturvedi [6] used a Ti-45Al-2Nb based alloy cast and HIPed, in which the heat treatments were performed in the α-phase field at a temperature of 1350 °C for periods of time varying from 2 min to 4 h using proper cooling rates to achieve a fully lamellar microstructure. Chen et al. [13] used an atomised and hot isostatic pressed Ti-48Al alloy consolidated at 1250 °C for 2 h and heat treated in the α-phase field at 1380 and 1400 °C for ½ and 1 h, respectively. In all research works, the authors unanimously agreed that the higher the annealing temperature is, the faster is the grain growth and long times of exposition enhance this effect; consequently, larger colonies resulted. Vanishing of the interlocking along the colony boundaries with increasing temperature was also observed.



(a) Reference sintered at 1500 °C for 2 h. Porosity of 0.65 ± 0.15%

(b) HT1 sintered at 1500 °C for 2 h followed by 3 h at 1350 °C. Porosity of 0.85 ± 0.15%



(c) HT2 sintered at 1500 °C for 2 h followed by 3 h at 1400 °C. Porosity of 0.50 ± 0.1%

Fig. 1. Light optical micrographs of sintered and heat-treated specimens. These images were taken from the centre of samples.

Typical room temperature tensile test curves are shown in **Fig. 2**. The heat treatments induced a decrease in the ultimate tensile strength and elongation in comparison to the Reference. However, HT1 and HT2 showed similar results, despite the significantly different colony sizes. Comparing the present results to those achieved by Limberg et al. [3] and Soyama et al. [4], in which the same alloy was processed under similar conditions, the values for ultimate tensile strength and elongation of the Reference sample are comparable, but HT1 and HT2 samples presented significantly lower values. Considering that the porosity levels of the specimens prepared in this study were in the same range, the microstructure changes caused by the different heat treatments seem to be the decisive factor causing the decrease in the mechanical properties.

The time until a specific amount of deformation is reached during creep testing is displayed in **Fig. 3**. The most creep resistant condition was the Reference, which took approximately 1.2 h to achieve 1% plastic strain followed by HT2 and HT1 with 0.9 and 0.75 h, respectively. It is noteworthy that the time until 1% strain reflects more or less the primary creep resistance [14].

In contrast to the classical creep theory, the specimens with larger colony sizes HT1 and HT2 showed poorer creep resistance. Nevertheless, it is important to point out that with the increase in colony size, a significant change in boundary morphology is associated. Consequently, it is possible that due to the decrease in interlocking between colonies, the primary creep strength also decreased. Additionally, the absence of interlocking has been reported to affect the creep strength negatively [6].

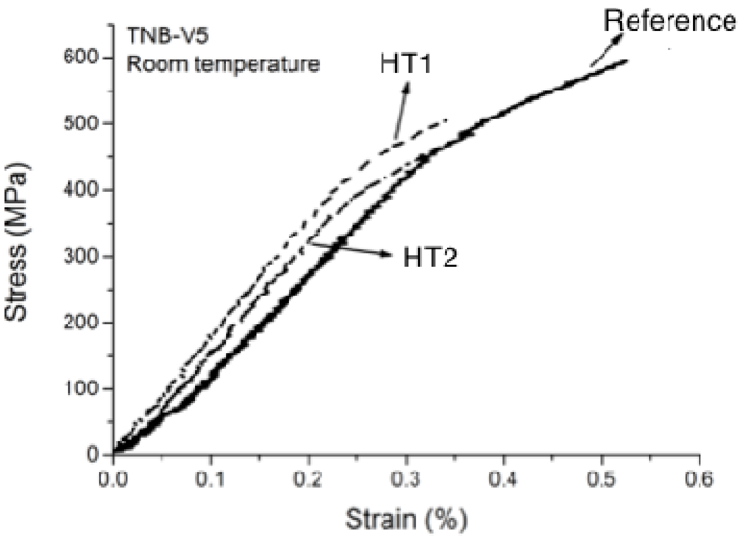


Fig. 2. Typical tensile test curves from each configuration measured at room temperature.

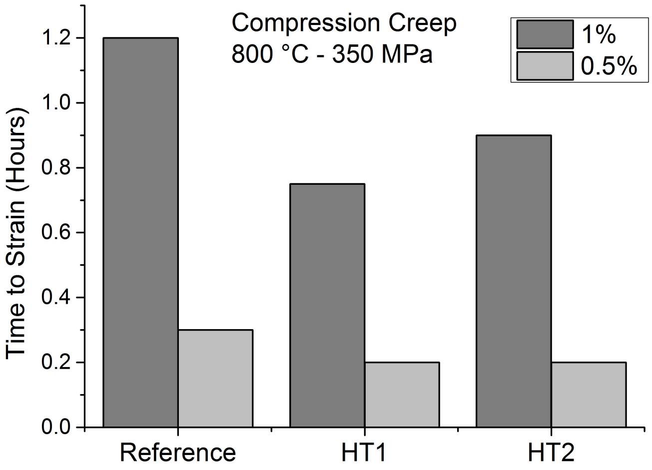


Fig. 3. Time for a specific amount of strain for each configuration under compression creep conditions of 800 °C and 350 MPa loading.

Contrary to the results from Maruyama et al. [8,9], at least for up to 1% of plastic deformation, colony sizes larger than 100 µm were detrimental for the creep resistance. In the present study, colonies smaller or close to 100 µm even exhibited improved creep strength up to a plastic deformation of 1%. Most probably this is no direct effect of the colony size but a beneficial effect of colony boundary interlocking. This is more pronounced in the specimens with smaller colony sizes. Therefore, the average colony size appears to be a parameter that not only controls tensile strength but also influences the creep resistance. It is difficult to determine if the colony size itself or colony boundary interlocking, which is associated with smaller colony size, causes the improvement in tensile strength. Nonetheless, it seems that the better creep strength is an effect of better colony boundary interlocking.

Conclusions

The effects of different heat treatments in the α-phase field after sintering in powder metallurgy processed TNB-V5 were investigated. Larger colony sizes were achieved after the heat treatments; however, their effect on tensile properties and primary creep resistance were unfavourable. In the case of creep deformation, this was probably due to the change in interlocking characteristics of the boundaries.

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