Microstructure and Mechanical Properties of Titanium Alloys with Bi-Lamellar Microstructures.

G. SCHROEDER, J. ALBRECHT, G. LÜTJERING
Technical University Hamburg-Harburg, 21071 Hamburg, Germany

ANNOTATION
The investment casting technology offers an attractive potential to reduce the manufacturing cost of a finished Ti-alloy part. Being a net-shape technology, however, the possibilities to modify the microstructure to improve the mechanical properties - or balance property combinations - are limited to purely thermal treatments. This paper presents results of an investigation aimed to probe the possibilities for property improvements by microstructural modification within the framework of thermal treatments. It could be shown, that the slip length, which is related to several mechanical properties, could be effectively reduced by a so-called "bi-lamellar" modification of the cast lamellar microstructure. During this treatment, fine α-plates are precipitated in widened β-"lamellae" hindering slip transfer. As a result, the yield stress, HCF-strength, resistance to microcrack propagation and to creep deformation are simultaneously improved while maintaining the resistance to fatigue crack propagation. A slight decrease of fracture toughness and a reduced ductility of the bi-lamellar conditions as compared to their lamellar counterparts were observed in Ti-6Al-4V, the alloy which is most widely used for casting applications.

Key words: Ti-6Al-4V, investment castings, microstructure, yield stress, fatigue resistance, crack propagation, creep resistance

1. INTRODUCTION
The bi-lamellar modification was designed to improve the properties of Ti-alloys with lamellar microstructures. The modification is a result of a purely thermal treatment, which makes it particularly interesting for being applied to investment castings. This net shape technology has in many cases a substantial potential to reduce the overall manufacturing cost of a titanium part [1], however, the possibility to modify the microstructure and thereby optimize or balance mechanical properties is greatly reduced compared to the forging route. During the forging sequence, optimized thermomechanical treatments can be easily incorporated, while for investment cast parts naturally only post-casting thermal treatments are possible.

The idea of the bi-lamellar heat treatment was to reduce the pile-up length by introducing obstacles to slip transfer. By an annealing treatment in the α+β phase field, the width of the β phase is increased, upon cooling, fine α platelets in different orientation variants are precipitated in the β phase, which effectively block cross-lamellar slip transfer.

Earlier investigations have shown [2, 3], that this modification has a potential to increase the yield stress, the fatigue strength and the creep resistance simultaneously. The processing parameters for the bi-lamellar heat treatment are the annealing temperature in the α+β phase field, and the cooling rate after the treatment. This paper focusses on the influence of the bi-lamellar heat treatment on the mechanical properties with emphasis on the fatigue behavior.

2. EXPERIMENTAL
The experiments concentrated on Ti-6Al-4V as the alloy most widely used for investment casting applications. Blanks with a size of about 70 x 70 mm and a thickness of 12 mm were prepared following the commercial route for investment casting, the final step being a HIP-treatment. In Ti-castings, the dimensions of the lamellar microstructure, particularly colony size and width of the lamellae, depend upon the cooling rate during casting,
which, for constant processing parameters, is determined by the wall thickness. To simulate a condition with a large wall thickness, blanks were heat treated for 30 minutes at a temperature in the β phase field (1050°C) and subsequently cooled with a cooling rate of 1°C/min.

For the bi-lamellar heat treatment, a temperature of 880°C was chosen on the basis of earlier investigations [2, 3]. The cooling rate after the annealing was varied from 15°C/min to 8000°C/min. The latter, very fast cooling rate was added strictly for scientific purposes. For real castings, the maximum cooling rate is limited by dimensional tolerances and residual stresses; an upper bound of about 600°C/min appears reasonable for parts with relatively simple geometries. After the bi-lamellar heat treatment, all specimens received an identical final aging treatment (24 h at 500°C) to precipitate fine Ti₃Al.

Besides tensile tests, S-N curves were measured at a R-ratio of -1, using hourglass shaped specimens with a round cross section, which were electrolytically polished prior to testing. The same type of specimens was used for investigating the microcrack propagation behavior at R = -1. Macrocrack propagation behavior was tested using compact tension specimens at R-ratios of 0.1 and 0.7. The investigations were completed by Kᵦ tests. Creep tests were performed at two different time/temperature combinations: i) at an applied (initial) stress of 400 MPa and at a temperature of 400°C where the plastic strain after a test period of 100 hours was evaluated, and ii) at an (initial) stress of 330 MPa and at a temperature of 350°C. In this case the plastic strain after 100 hours and after 500 hours was evaluated. This latter combination is more realistic in terms of practical applications, however, the resulting plastic strains even after 500 hours are so small that it is difficult to identify differences caused by modifications in the microstructure. At higher stress (400 MPa) and higher temperature, these differences become more obvious.

3. RESULTS AND DISCUSSION

The microstructures of selected bi-lamellar conditions are shown in the TEM-micrographs Fig. 1 in comparison with the coarse lamellar microstructure (cooling rate during casting: 1°C/min). It is clearly seen that the width of the β phase has increased due to the annealing treatment and that α-platelets are precipitated in the β phase. With the exception of the condition cooled at the very slow rate of 15°C/min, the width of the hardened β lamellae is fairly constant for all bi-lamellar conditions, however, with increasing cooling rate, the size of the α-platelets within the β-lamellae decreases continuously. At the highest cooling rate of 8000°C/min, the platelets result from a martensitic transformation rather than from diffusionally controlled growth (slower cooling rates). For the very slowly cooled condition, the width of the transformed β phase is smaller due to shrinkage during slow cooling concomitant with the precipitation of α plates.

The yield stress values are shown in Fig. 2 as a function of cooling rate after the bi-lamellar heat treatment. The value for 1°C/min corresponds to the lamellar reference condition, which has a yield stress of 810 MPa. For the bi-lamellar conditions, the yield stress increases continuously with increasing cooling rate. The highest increase in yield stress to a value of 980 MPa is found for the fastest cooling rate (8000°C/min), but even in the range of cooling rates which are of technical interest (up to 600°C/min), an improvement of 100 MPa is feasible. This increase in yield stress is a result of the reduction of the effective pile-up length by the bi-lamellar modification. The increase of the yield stress with increasing cooling rate is a result of the decreasing size of the α-platelets, i.e. increasing hardness of the modified β-lamellae.

With increasing yield stress, the ductility decreases, as shown in Fig. 3. The tensile elongation decreases from about 11% for the lamellar condition to 4.5% for the bi-lamellar condition with the fast cooling rates. For all conditions, lamellar as well as bi-lamellar, the fracture mode is ductile (dimple type). Fig. 4a shows a fractograph of the lamellar condition, exhibiting a completely transgranular, ductile fracture across colonies. The bi-lamellar condition with the fastest cooling rate (8000°C/min), however, shows large areas of intergranular fracture (Fig. 4b), which is microscopically ductile, as shown by the dimples on the facets. This observation suggests a change in fracture mode from a transgranular, cross-colony type for the lamellar condition to a fracture along GB-α layers. This change is a result of the increased hardness of the matrix for the bi-lamellar conditions, leading to preferential plastic deformation of the softer GB-α layers and subsequent cracking along these layers [4]. This change in fracture mode explains the lower ductility of the bi-lamellar conditions.

The high-cycle fatigue strength of the bi-lamellar conditions is improved in comparison to the lamellar condition (Fig. 5), following the trend found for the yield stress (cf. Fig. 2). This is a commonly observed correlation; the high cycle fatigue strength, typically at 10⁷ cycles, reflects the material's resistance to crack nucleation, resulting from dislocation activity. Hindering dislocation motion generally delays fatigue crack nucleation and simultaneously increases the yield stress [5]. Fig. 5 also shows the curve for the fatigue resistance at 10⁷ cycles, indicating that the LCF behavior of the bi-lamellar conditions is also superior to the lamellar condition. This leads immediately to the propagation behavior of microcracks since in the LCF-range about 50% of the total life is spent in the stage of crack nucleation, the remaining 50% in the stage of microcrack propagation, while the contribution of macrocrack propagation is negligible.

The da/dN-ΔK curves for microcrack propagation are shown in Fig. 6. The highest crack propagation rates are
Fig. 1: Microstructure (TEM)
a) lamellar
b) - e) bi-lamellar with different cooling rates

d) 600°C/min

e) 8000°C/min

Fig. 2: Yield Stress comparison
1°C/min: lamellar, others: bi-lamellar

Fig. 3: Ductility comparison
1°C/min: lamellar, others: bi-lamellar

Fig. 4: Change in tensile fracture mode
a) lamellar: cross-colony
b) bi-lamellar (8000°C/min): along GB-α layers
Fig. 5: Fatigue strength ($10^7$ and $10^9$ cycles)
$1^\circ$C/min: lamellar, others: bi-lamellar

Fig. 6: Comparison of microcrack propagation behavior

Fig. 7: Microcrack propagation
a)-b) lamellar
c)-d) bi-lamellar ($70^\circ$C/min)
found for the lamellar condition. For the bi-lamellar conditions, the cooling rate after the annealing treatment shows no significant effect. The lower crack propagation rates of the bi-lamellar conditions are a result of the high density of obstacles, which the growing crack encounters in the form of hardened β lamellae.

Microcracks are typically nucleated across colonies in the lamellar as well as in the bi-lamellar conditions. A typical example is shown in Fig. 7 (lamellar). Fig. 7a shows a very small crack, which was detected after 5000 cycles, Fig. 7b shows the extension of this crack within one colony after an additional 1000 cycles. Crack nucleation was also observed at colony boundaries for the lamellar condition as well as for the bi-lamellar conditions. Examples are shown in Fig. 7c (N = 10000 cycles) and Fig. 7d (N = 12000), where the crack nucleated at a colony boundary and propagated at a later stage across adjacent colonies (Fig. 7d).

Fig. 8 shows the fatigue crack propagation behavior of macrocracks at a R-ratio of 0.1. In the threshold regime, the propagation rate of the macrocracks for all conditions is more than two orders of magnitude lower than that of the microcracks (cf. Fig. 6). This demonstrates the strong effect of the crack front geometry, which develops with increasing crack front length. The differences in crack propagation rate of the macrocracks in the threshold regime is quite small for the conditions tested, except for a slight tendency of the bi-lamellar condition with the fastest cooling rate (8000°C/min) to be at the upper end of the scatter band. Investigations of the crack front profiles showed (Fig. 9), that the bi-lamellar condition with the fastest cooling rate has a somewhat flatter crack front profile (Fig. 9a) than the lamellar condition (Fig. 9b).

Measurements of crack closure showed, that the bi-lamellar condition (8000°C/min) had a distinctly higher degree of closure (35% at a da/dN of 10⁻⁴ m/cycle) than the lamellar condition (17% at a da/dN of 10⁻⁴ m/cycle), despite of the flatter crack front profile of the bi-lamellar condition (cf. Fig. 8). The phenomenon is explained by a pronounced difference in the fracture surface roughness of the two conditions on a microscopic scale. As shown in the fractographs Fig. 10, on the scale of the lamellar dimensions, the fracture surface of the lamellar condition is fairly smooth. For this condition, the width of β-lamellae is quite small and the hardness of the α- and of the β-lamellae is similar, resulting in a fairly smooth fracture surface within one colony (Fig. 10a). In the bi-lamellar condition (8000°C/min), however, the hardened β-lamellae are distinctly wider and their hardness is considerably higher. Due to the higher hardness of the transformed β-lamellae, cracks formed in the α-lamellae try to extend along the interface between the α- and the hardened β-lamellae for a short distance, leading to small steps, as shown schematically in Fig. 10b and in the fractograph Fig. 10c. With the presence of these small steps, only slight shear displacements (mode II) at the crack tip are required to cause crack closure. This retarding contribution of closure counteracts the effect of the flatter crack front profile for this bi-lamellar condition. This is demonstrated in Fig. 11, which shows the crack propagation rates as a function of the closure corrected ΔK-values (ΔK_{corr}). In this representation, the bi-lamellar condition with the fastest cooling rate shows distinctly higher crack propagation rates in the threshold regime than the other conditions, as indicated by the flatter crack front profile.

The same result is found for tests, where the R-ratio was raised to 0.7 to prevent crack closure. In this situation, the fast-cooled bi-lamellar condition shows noticeably higher propagation rates in the threshold regime (Fig. 12). It should be emphasized, that a difference in fatigue crack propagation rates is only evident for a bi-lamellar condition with the very fast cooling rate of 8000°C/min. Within the range of technically more relevant cooling rates (up to 600°C/min), the fatigue crack propagation behavior of the bi-lamellar conditions is nearly identical to that of the lamellar condition.

To complete the comparison of the crack propagation behavior of lamellar and bi-lamellar conditions, fracture toughness tests were performed. Since the thickness of the specimens was limited by the dimensions of the cast plates to 10 mm, it was not possible to establish ASTM-valid K_{lc} values. The comparison of the K_{max}-values (Fig. 13) for the lamellar and the bi-lamellar conditions shows a tendency for slightly decreasing fracture toughness values of the bi-lamellar conditions as compared to the lamellar condition. This slight decrease in fracture toughness is believed to be due to the somewhat flatter crack front profile of the bi-lamellar conditions.

With respect to creep resistance, for many applications the plastic strain after 100 or 200 hours is technically more relevant than fracture related creep properties. Fig. 14 shows the plastic creep strain after 100 hours for the high test temperature (400°C) as a function of cooling rate from the bi-lamellar heat treatment temperature (1°C/min corresponds to the lamellar condition). At these creep conditions, the lamellar condition shows a plastic strain of 0.24%, while the bi-lamellar conditions show distinctly lower values with a minimum of 0.12% at a cooling rate of 200°C/min. A similar tendency is found for the lower test temperature of 350°C (Fig. 15), where the applied stress was also reduced from 400 MPa to 330 MPa. Naturally, for all conditions the creep strains are quite small even at the extended creep time of 500 hours, but there is a clear tendency for the bi-lamellar condition cooled at a rate of 600°C/min to have a lower creep strain than the lamellar condition, particularly after a test period of 100 hours. With prolonged creep time, the plastic creep strains for the three conditions are comparable.
Fig. 8: Propagation behavior of macrocracks at $R=0.1$

Fig. 9: Crack front profiles (CT-specimens)
- da/dN=10^{-9}m/cycle
  - a) lamellar
  - b) bi-lamellar ($800^\circ C/min$)

Fig. 10: Fracture surfaces, Fatigue crack propagation at $R=0.1$; da/dN=10^{-9}m/cycle
- a) lamellar, 17% closure; b) step formation, schematically; c) bi-lamellar ($800^\circ C/min$), 35% closure

Fig. 11: da/dN ($R=0.1$) as a function of closure corrected $\Delta K$

Fig. 12: Propagation behavior of macrocracks at $R=0.7$
Another subject of technical relevance is the primary creep regime. Fig. 16 shows the creep rate versus the creep strain for the creep conditions 400°C and 400 MPa. It is clearly seen that the creep rate in the primary creep regime is much lower for all bi-lamellar conditions as compared to the lamellar condition. The creep rate in the steady state regime, however, is quite similar for all conditions, which suggests that the steady-state creep mechanism is the same for lamellar and bi-lamellar microstructures. Consequently, the differences in creep strain after 100 hours shown in Fig. 14 are largely due to the improved creep resistance of the bi-lamellar conditions in the primary creep regime. This finding is supported by the behavior at 350°C, 330 MPa, shown in Fig. 17. Again, the creep rates of the bi-lamellar conditions in the primary creep regime are much lower compared to the lamellar condition. After about the same creep strain of 0.04%, all conditions reach a similar steady-state creep rate. The improved creep behavior of the bi-lamellar conditions in the primary creep regime is attributed to the effective hardening of the β phase. In (α+β) Ti-alloys, the deformation of the β phase plays an important role under
dislocation controlled creep conditions. The precipitation of the fine α platelets in the β phase reduces the plastic deformation in the β "lamellae" and thereby also the overall creep strain.

4. CONCLUSIONS

The investigation showed, that the coarse lamellar microstructure typically found in investment castings of Ti-6Al-4V can be modified by a simple heat treatment in the (α+β) phase field. As a result of this annealing treatment, the width of the β-"lamellae" is increased, and during subsequent cooling, fine α platelets in different orientation variants can be precipitated in the β-phase. It was shown, that this so-called "bi-lamellar" modification has a potential to improve several important mechanical properties.

In particular:
- The yield stress is improved over the lamellar ("as-cast") condition due to the reduction of the slip-length (no slip-transfer within one colony) and the effective hardening of the β-phase.
- The high-cycle fatigue strength of the bi-lamellar conditions is higher as compared to the lamellar conditions due to the increase in yield stress (delayed crack nucleation).
- Microcrack propagation is slower in the bi-lamellar conditions due to the high density of obstacles (hardened β-"lamellae") retarding crack growth.
- The low-cycle fatigue resistance is positively influenced by both the delay in crack nucleation and the retardation of the growth of microcracks.
- The propagation behavior of macrocracks at low R-ratios as well as at high R-ratios is similar in lamellar and in bi-lamellar microstructures.
- The fracture toughness of the bi-lamellar microstructures appears to be slightly lower than that of the lamellar condition, due to the slightly flatter crack front profile of the bi-lamellar conditions.
- The ductility of the bi-lamellar conditions is lower than that of the lamellar condition. This is caused by a change in fracture mode from a cross-colony dimple type of fracture (lamellar) to a microscopically ductile fracture along GB-α layers due to an increasing hardness of the bi-lamellar matrix.
- The creep resistance of the bi-lamellar conditions is higher as compared to the lamellar condition due to the effective hardening of the β-phase.

The results indicate, that the magnitude of the improvements, particularly in yield stress and HCF-resistance, is related to the cooling rate from the bi-lamellar heat treatment temperature, i.e. yield stress (and HCF-strength) increase with increasing cooling rate. For practical applications of this treatment to cast parts, the maximum cooling rate is limited by the complexity of the geometry of the part. With respect to the processing steps during investment casting, the bi-lamellar heat treatment should be performed right after hot-isostatic pressing (HIP), routinely applied for parts for critical applications (aircraft industry). In this case, the treatment could be done in a normal furnace rather than in vacuum or inert atmosphere, allowing a wider range of cooling rates at lower cost. Surface oxide (α-case) would be removed during the subsequent pickling treatment.

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6. REFERENCES