Microstructural Characteristics and Creep Behavior of 45XD TiAl Alloys*

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A near lamellar microstructure and two fine-grained fully lamellar (FGFL) microstructures in Ti-45Al-2Nb-2Mn+0.8 vol%TiB2 alloy were prepared by selected heat treatments, and the fully lamellar microstructures were aged for stabilizing the lamellar plates. Microstructural examination and tensile creep tests at 760 °C showed that the near lamellar microstructure possessed inferior creep resistance due to its coarse lamellar spacing and its larger amount of equiaxed γ grains at colony boundaries. The fine lamellar spacing as well as the fine lamellar colony size gave a major contribution to make the minimum creep rates smaller in the fully lamellar TiAl alloys. Since aging treatments stabilized the lamellar microstructures and delayed the degradation process during creep deformation, the aged samples exhibited lower minimum creep rate and longer creep life than the corresponding samples without the aging treatment. These results suggest that a fine as well as stabilized fully lamellar structure is a critical factor to improve the creep resistance of TiAl alloys in terms of short and long-term creep.

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1. Introduction

TiAl alloys are appealing for use in elevated temperature structural applications because of their low density, high specific strength, high specific stiffness and good high-temperature strength. However, the poor ductility of TiAl alloys at ambient temperature has limited their applications.1) To overcome this obstacle, some developments of TiAl alloys have been focused on refining the lamellar microstructure.1–3) Adding boron has been proved to be an effective way to refine the as-cast microstructure of TiAl alloys.2) The lamellar microstructure can also be refined by appropriate heat treatment.4) Investment cast XD alloys have uniform cast microstructures with relatively fine grain sizes. Microstructural responses to various heat treatment conditions in the alloys have been investigated systematically.4) However, limited work has been reported on the microstructural effect on creep properties.

In this study, a near lamellar microstructure and two FGFL microstructures in 45XD alloy were prepared by appropriate heat treatments. To stabilize the FGFL microstructures, selected aging treatments were added. The creep behaviors derived from these microstructures were compared. The influences of their microstructural factors involving colony size, lamellar spacing and volume fraction of lamellar colony, and microstructural stability on the creep behavior were discussed.

2. Experimental Procedure

Investment cast bars with nominal composition of Ti-45Al-2Nb-2Mn+0.8 vol%TiB2 (at%) (45XD) were used in this study. The cast bars were hot isostatically pressed (HIP) at 1260 °C and 175 MPa for 4 hours to remove cast porosities prior to the following heat treatments. In order to control various microstructures, three kinds of heat treatment conditions were selected. The first group of samples was heat treated at 1010 °C for 50 hours and then air-cooled (AC1). The second group of samples was heat treated at 1350 °C for 0.5 hours and then air-cooled (AC2). The third group of samples was heat treated at 1350 °C for 2 hours followed by oil quench (OQ1). For stabilizing the microstructures, the samples heat-treated by AC2 were aged subsequently at 1000 °C for 8 hours (AC3), and the samples heat-treated by OQ1 were subjected to multiple-step aging (OQ2). For all the heat treatment procedures, the samples were wrapped in Ta foil and sealed in quartz tubes back-filled with Ar to 220 mm Hg.

Creep samples were machined by low stress grinding to a straight gauge length of 22 mm and a diameter of 4 mm. Creep samples were tested in tension in air at 760 °C at a constant load of 207 MPa. Creep strain was measured with an LVDT equipped extensometer attached to grooves in the sample shoulders, providing strain resolution of at least ±1.5 × 10−5.

For microstructural examination, the heat-treated and the creep samples were polished electrolytically in a solution of 64% methanol +31% butanol +5% perchloric acid at −25 °C. For analysis of different phases, the electropolished samples were examined in a Philips XL30S SEM at 20 kV, using the backscattered electron (BSE) technique. For TEM examination, thin foils were thinned by twin-jet polishing with the electrolyte at −45 °C. TEM analyses were carried out on a JEOL EX 200 microscope operating at 200 kV.

3. Results and Discussion

The microstructure after heat treatment of AC1, in Fig. 1(a), has a mixture of predominant lamellar colonies and relatively small amount of equiaxed γ grains, namely near lamellar structure, which is also called standard microstructure for 45XD alloy. The volume fraction of lamellar colony is more than 80%, a colony size is about 20 to 100 μm, and the lamellar spacing is relatively coarse (Fig. 1(a)). Heat treatments at both AC2 and OQ1 produce fully lamellar...
structures (Figs. 1(b), (c)). Grain boundary morphologies in the air-cooled conditions are serrated. The colony size and lamellar spacing of OQ1 sample is in the range of 10 to 50 μm and about 0.04 μm, respectively. Unlike the air-cooled condition, most of the grain boundary morphologies are planar types in the oil quench condition (Fig. 1(c)).

In Fig. 2, SEM-BSE images of AC3 and OQ2 samples show that near the lamellar colony boundary, a large amount of γ phases are observed in both conditions compared to the unaged conditions (Fig. 1(b), Fig. 1(c)). Especially, in AC3 samples, coarse equiaxed γ grains are formed due to recrystallization (see arrows in Fig. 2(a)). However, in OQ2 condition, grain boundary morphology is almost same as the unaged condition, indicating no recrystallization of γ grains (Fig. 1(c) and Fig. 2(b)).

Figure 3 shows lamellar microstructure within the lamellar colony before and after aging in OQ samples. It is clear that α₂ lamellae become thinner and γ lamellae become coarser while the γ/α₂ interfaces keep their continuity (arrows in Fig. 3 indicate α₂ lamellae). Therefore, it is speculated that the aging treatments result in phase transformation of α₂ to γ, which leads to less volume fraction of α₂ phase due to relatively unstable lamellar structure in the unaged condition (AC2 and OQ1). Given this result, the phase transformation during aging makes the lamellar microstructure closer to phase equilibrium condition and more stable, which leads to formation of equiaxed γ grains at the lamellar colony boundary and/or coarsening of γ lamellae within the lamellar colony depending on the condition of aging.

Creep curves for the unaged and variously aged samples are drawn in Fig. 4. Creep strain rate decreases rapidly until it reaches a minimum value, and then the creep strain rate increases gradually to fracture with strain, giving an extended
period of creep life. The standard microstructure exhibits the largest minimum creep rate ($3 \times 10^{-4}$ h$^{-1}$) and the shortest creep life (290 hours), due to coarse lamellar spacing and a larger amount of $\gamma$ grains at the colony boundaries. The fully lamellar structures, especially in the aged conditions, show superior creep properties. For example, the minimum creep rates of the AC1 and the OQ2 are $1 \times 10^{-4}$ h$^{-1}$ and $1 \times 10^{-3}$ h$^{-1}$, respectively. In addition, creep life in the AC3 sample is about 450 hours, which is almost 60% increase compared to the life of the sample with the standard microstructure.

From previous work, it is found that the creep rate is independent of colony size when it is larger than 100 $\mu$m, and colony size can be reduced to 100 $\mu$m without losing creep resistance. If grain boundaries are unstable, colony size may affect creep rate when the colony size is relatively small. The colony sizes of the fully lamellar microstructures in AC2 and AC3 samples are less than 100 $\mu$m, and the OQ produces fine colony size less than 50 $\mu$m. However, as indicated above, the microstructure in the OQ1 condition is relatively unstable. Unlike standard and air-cooled microstructures (AC1 and AC2), although colony boundary morphology is a planar type in the OQ conditions, much finer lamellar spacing and colony size result in lower minimum creep rate. Previous study shows that the creep rate decreases with decreasing lamellar spacing. Therefore, finer lamellar spacing would be a major factor to cause lower minimum creep rate, and finer colony size might also contribute to lower creep strain rate. Although more $\gamma$ phases near colony boundaries are formed after aging, the aged samples show better creep resistance than the corresponding samples without the aging. Therefore, stabilization of lamellar microstructure is important for enhancement of creep resistance (creep life), while the fine lamellar spacing and colony size are essential if primary creep resistance is concerned. From previous work, the $\gamma$ phase gives the lower creep rate than the $\alpha_2$ phase, and a higher volume fraction of $\gamma$ phase gives a lower creep rate if the rule of mixture holds. However, if the volume fraction of the $\gamma$ phase is increased by increasing Al concentration, the creep rates of lamellar TiAl alloys are insensitive to the volume fraction of constituent phases because of less $\gamma/\alpha_2$ interface with thick $\alpha_2$ lamellae as described in the previous work. In contrast to this, if the volume fraction of $\alpha_2$ lamellae is reduced by aging treatment without reducing the number of $\gamma/\alpha_2$ interface, such as by the multiple aging (OQ2), the creep rate should decrease with decreasing the volume fraction of $\alpha_2$ lamellae.

During the creep deformation, the degradation of the microstructure occurs because of the applied stress and the thermal instability as shown in Fig. 5. From examination of deformed microstructures of the AC2 and AC3 samples crept near the tertiary creep stage, spheroidization within lamellae and recrystallization of $\gamma$ grains at lamellar colony boundaries are supposed as the major degradation processes in the AC2 sample, forming fine $\gamma$ globular structures at the lamellar colony boundary regions (Fig. 5(a)). Because of strong orientation dependence on deformation mode, extensive local deformation would be expected at the fine globular colony boundary region, where various oriented

**Fig. 4** (a) Creep curves and (b) creep rate versus strain curves of 45XD TiAl alloy after various heat treatments.

**Fig. 5** SEM-BSE images showing cavity nucleation of 45XD TiAl alloy in the tertiary stage (a) 1350 C/0.5h/AC (b) 1350 C/0.5h/AC+1000 C/8h/AC.
lamellar colonies are existed. Therefore, the incompatible deformation results in nucleation of voids at the lamellar colony boundary regions where local deformation exceeds a limit value (Fig. 5(a)). In the AC3 sample, the degree of microstructural degradation is much less than the AC2 due to stabilized microstructure by aging, but similarly, voids are nucleated and formed at the colony boundary (Fig. 5(b)). With further deformation, the degradation processes proceed, and the voids propagate along the colony boundary and coalesce until fracture (Fig. 6). The fine globular structures are also found in the colony boundary region in the AC3 sample after fracture (indicated by arrow in Fig. 6(a)). Therefore, the aging treatment delays the degradation processes with some degree, which extends the tertiary stage and results in lower minimum creep life of the AC3 sample. The degradation process at the colony boundary for the OQ2 sample (Fig. 6(b)) is more severe by comparison to the AC3 sample, which might be responsible for finer lamellar colony size rather than finer lamellar spacing. This fact accords with the faster tertiary creep rate of the OQ2 sample than that of the AC3 sample. It is concluded that finer lamellar spacing and colony size may result in lower minimum creep rate and slower creep process before onset of tertiary stage, while finer lamellar colony size is detrimental to tertiary creep unless the lamellar structure is fully stabilized.

4. Summary

In 45XD TiAl alloys, the standard microstructure shows inferior creep resistance to the modified fully lamellar structures. Fine lamellar spacing as well as fine lamellar colony size gives a major contribution to lower minimum creep rates in fully lamellar TiAl alloys. Aging treatments stabilize the lamellar structure to a certain extent and delay the degradation process although microstructural changes such as coarsening of γ lamellae within lamellar colony and formation of fine globular structures at lamellar colony boundaries occur during aging. As a result, the aged samples exhibit lower minimum creep rate, and longer creep life than corresponding samples without the aging treatment. During creep deformation, degradation of the microstructure occurs, and voids nucleate at lamellar colony boundaries and propagate along the boundaries until fracture. The microstructure stability influences not only the minimum creep rate, but also the creep life. Therefore, a fine as well as stabilized fully lamellar structure is a critical factor to improve the creep resistance of TiAl alloys in terms of short and long-term creep.

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