Analysis of creep behaviour of TiAl-8Ta intermetallic alloy

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Abstract. The creep behaviour of a high Ta containing γ-TiAl alloy was investigated in the temperature range of 700-850°C with applied stresses in order to have rupture times up to 3000 h. Decelerating primary and accelerating tertiary regimes dominated the experimental curves, whilst the secondary regime with constant minimum creep rates was absent. Reporting the experimental data in plot log $\dot{\varepsilon}$ vs. $\varepsilon$, the tertiary data lie on lines with similar slopes at all temperature and load conditions, indicating that a damage mechanism, depending only on the accumulated creep strain causes the accelerating tertiary regime. Creep tests with step like changes in load and/or temperature changes were run and microstructure investigations were performed through X ray diffraction, scanning and transmission electron microscopy to have an insight on the nature of the damage mechanisms that control the accelerating tertiary regime.

Introduction

The dual phase $\alpha_2 + $ γ TiAl alloys have been widely investigated, since they are candidates to be used in aeronautic and land based gas turbine [1-3]. A route to produce the desired fine microstructure in TiAl alloys rich in heavy elements such as Nb and Ta is based on the massive transformation typical of such alloys [4-7]. The designed thermal and HIP treatments, following to the massive transformation, are imposed to obtain the final convoluted cross $\alpha_2 + $ γ lamellar microstructure that confer to the alloys an adequate room temperature ductility, the critical parameter for intermetallic alloy applications.

The physical and mechanical proprieties of a new Ti-46Al-8Ta (atom. %) have been studied in the integrated European project IMPRESS. In the present work the first creep characterisation results are reported with particular attention on the accelerating tertiary regime that dominates the creep curves of TiAl-8Ta, and on the damaging mechanisms that cause the accelerating regime through creep tests with changing conditions and microstructure investigations.

Material and experimental procedure

The alloy Ti-46Al-8Ta (atom. %) was provided by ACCESS (Aachen) in the form of cast and heat treated cylindrical bars with a diameter of 13 mm and length of 120 mm. Heat treatment consisted of hot isostatic pressing (HIP) at an applied pressure of 200 MPa, temperature of 1260 °C for 4 h, which was followed by solution annealing at 1360 °C for 1 h and air cooling. A final HIP ageing at an applied pressure of 150 MPa and temperature of 1260°C was imposed for 2 h, followed by cooling at a rate of 5 °C/min.

Constant load creep tests were performed on cylindrical samples with gauge length and diameter of 28.0 and 5.6 mm respectively. The gauge elongation was measured through capacitive transducers connected to extensometers clamped to the specimen gauge ledges. The specimens were heated through furnaces and the gauge temperature was controlled through three thermocouples in order to avoid temperature gradients along the specimen gauge.

The microstructure characterization was carried out through X ray diffraction (XRD), scanning electron microscopy (SEM) with energy dispersion spectroscopy (EDS) and transmission electron microscopy (TEM). The TiAl-8Ta specimens for SEM were polished and were not etched chemically, since the backscattered electron signal in SEM was adequate to collect quality micrographs.
Results and discussion

3.1. Creep test results

When the true creep rates $\dot{\varepsilon}$ are plotted against the true creep strains $\varepsilon$, the TiAl-8Ta curves do not exhibit a wide plateau corresponding to the conventional secondary regime, but decelerating primary and accelerating tertiary regimes dominate the experimental curves at all the present experimental conditions. Minima of the creep rates, i.e. $\dot{\varepsilon}_{\text{min}}$, are observable rather than steady state creep rate regimes. From the analysis of log $\dot{\varepsilon}_{\text{min}}$ vs. log $\sigma$ plots, where $\sigma$ is the nominal applied stress, different Norton exponents were obtained at different explored temperatures: 7.6 at 700°C, 6.6 at 750°C, 6.3 at 800°C and 4.9 from 850°C.

In log $\dot{\varepsilon}$ vs. $\varepsilon$ plot, the experimental points representing the long accelerating creep, lie on straight lines with similar slopes at all tested temperatures and loads. Therefore, the tertiary creep can be described by the following relationship:

$$\dot{\varepsilon} = \dot{\varepsilon}_0 \exp(m\varepsilon)$$

where $\dot{\varepsilon}_0$ is the back-extrapolated strain rate to $\varepsilon = 0$ and $m = 15.6 \pm 1.7$. In the log $\dot{\varepsilon}$ vs. $\varepsilon$ plots, the experimental creep curves are congruent, i.e. it is possible to overlap the creep curves obtained at different stresses and temperatures by a translation along the $\dot{\varepsilon}$ axis. As a consequence, the shape of the creep curves is independent on the applied stresses and all the creep curves coincide if plotted as strain in function of the fraction life $t/t_f$ where $t_f$ is the time to fracture or, the time to reach a defined strain. The experimental results indicate that the damage producing the dominant accelerating stage depends on strain only and the creep strain univocally quantify both the damage and the life fraction of the material. Such a behaviour is generally not found in nickel base superalloys, where there is a systematic variation of the creep curve shape at different stresses [8].

In order to confirm that the damage producing the accelerating creep is independent on the applied stress and on the time spent at high temperature, creep tests with step like load changes were run at 850°C when the microstructure degradation was expected to be more significant. The alloy was first loaded with an initial stress of 200 MPa to about 5%, corresponding at about half of the creep life, after that the nominal stress was reduced to 150 MPa. A second test consisted of 200 MPa to about 5%, after that a period of 500 h in temperature with no applied load was imposed and finally a nominal load of 150 MPa was applied. The experimental results of the variable load creep tests between 200 and 150 MPa are plotted as log $\dot{\varepsilon}$ vs. $\varepsilon$ in figure 1 and confronted with the constant load creep curves: following the stress increment and after a small and short transient, the strain rates lie over the reference constant load creep curves at the final applied stress. A further test consisted of 150 MPa to about 5%, after that an increase of nominal load to 200 MPa was imposed. The data are reported in log $\dot{\varepsilon}$ vs. $\varepsilon$ and compared to the 200 MPa + 150 MPa test in figure 2.
again, after reloading, no significant change of the strain rates and the tertiary creep slopes occurred. The experimental results confirm the damage is not depending on time and on applied stresses, but only on the accumulated creep strain. Moreover, the small and short transient following the reloading, after a long permanence at high temperature without load, show the static recovery of the dislocation microstructure is very low. The average of the activation volumes calculated through the above stress change creep tests resulted in about $6.9b^3$, where $b = 0.283$ nm with the Taylor factor assumed to be 3.06 [9].

Figure 3. TiAl-8Ta microstructure after creep at 700°C: cross lamellar $\gamma + \alpha_2$ structure similar to the as received structure ($\alpha_2$ bright). The grain boundaries present numerous Ta rich precipitates

Figure 4. TiAl-8Ta microstructure after creep at 850°C: the $\alpha_2$ lamellae are reduced in number and thickness; the grain boundaries present Ta rich precipitates and are depleted in Ta.

Figure 5. TEM micrograph of TiAl-8Ta after creep at 850°C. Dark thin lamellae are $\alpha_2$ phase and the bright phase is $\gamma$: cut of $\alpha_2$ phase is evident.

Figure 6. TEM micrograph of TiAl-8Ta after creep at 850°C. The planes of cut in $\alpha_2$ phase are put in evidence.

Also creep tests with change of temperature from 850°C to 800°C and from 750°C to 700°C with constant load shown the same behaviour. The apparent activation energy of deformation resulted to be 356.5 kJ·mol$^{-1}$ at 700°C and 365.4 kJ·mol$^{-1}$ at 850°C, in both cases similar to the apparent energy for Al diffusion in Ti about 350 kJ·mol$^{-1}$ in [8]. The found apparent energy of deformation and activation volume indicate that creep deformation in TiAl-8Ta in the present testing conditions is controlled by a dislocation climb [9].

3.2. Microstructure analysis

The microstructure of the alloy was examined before and after creep tests. Micrographs of the crept microstructure after about 1100 h at 700°C with nominal applied stress of 400 MPa and after about 500 h at 850°C/150 MPa are reported in figure 3 and 4 respectively. The microstructure of the crept alloy at 700°C is not significantly evolved with respect to the as received conditions, where cross lamellar $\gamma + \alpha_2$ structure is
observable. Conversely after creep at 850°C the microstructure is considerably evolved with respect to the as-received material: the $\alpha_2$ lamellae are reduced in number and thickness, a second phase rich in Ta precipitates profusely at the grain boundaries and the grain boundaries appear dark because of Ta depletion. Similar grain boundary degradation has been reported in TiAl alloys with significant addition of Nb [10]. The Ta rich phase was observed also in the as received alloy and at 700°C, however in smaller quantity. The XRD analysis confirmed the reduction of $\alpha_2$ volume fraction at 850°C and indicated that the Ta rich phase was the ternary phase Ti$_4$TaAl$_3$, designated as $\tau$ with hexagonal crystal structure of the B8$_2$ [11]. At all the explored test conditions no sign of cavitations was detected.

Selected TEM micrographs of the TiAl-8Ta alloy crept at 850°C with an initial load of 100 MPa crept to rupture in about 3500 h are reported in figures 5 and 6. Several $\alpha_2$ lamellae are still observable and they appear to be significantly cut, as shown in figure 6, where the planes of cut is put in evidence. The TiAl-8Ta crept at 700°C presented similar microstructure. The alloy did not present any sign of mechanical twinning that is very common in TiAl alloys [12-14].

The significant $\alpha_2$ volume fraction reduction and grain boundary degradation during creep, particularly important in the tests performed at the highest temperatures, do not seem to be able to describe the experimental accelerating creep in all the temperature and stress conditions investigated. Furthermore, cavity formation at the grain boundaries, significant in other alloys for high temperature applications [8], has not been reported in the present alloy. A microstructure damage mechanism that is sensitive to strain only is not evident from microstructure results and further investigation is needed.

Conclusions

The experimental investigation on the creep behaviour of a high Ta containing $\gamma$-TiAl alloy has shown:

- the creep curves are dominated by the tertiary accelerating creep stage and no steady state was observed;
- the apparent activation energy of creep and activation volume indicate that creep deformation in TiAl-8Ta is controlled by a dislocation climb;
- the increment of the strain rate with the strain during the accelerating follows the same exponential strain softening law in all the explored temperature and stress conditions, in spite of widely differing rupture life;
- the accumulated creep strain is a good parameter to quantify the damage of the alloy;
- mechanical twinning has not been detected;
- the significant $\alpha_2$ volume fraction reduction and grain boundary degradation during creep, specially in the tests performed at the highest temperatures, do not seem to be able to describe the experimental accelerating creep.

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