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The effect of environment and structure on creep fatigue crack propagation in a P/M astroloy

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The effect of environment and structure on creep fatigue crack propagation in a P/M Astroloy has been investigated by performing tests in air and in vacuum at 650°C and 750°C. Complex mechanical and environmental interactions have been revealed at 750°C temperature at which the $da/dN-\Delta K$ relationship obtained in air appears to be highly dependent of the initial ΔK values applied at the beginning of the creep-fatigue test. The effect of grain size with smooth grain boundaries appears to be strongly related to environmental effects rather than to creep properties. A serrated grain boundary morphology considerably improves crack growth resistance at 750°C under creep-fatigue conditions.

INTRODUCTION

Nickel base superalloys, elaborated by powder metallurgy are used for the manufacturing of turbine discs employed up to about 650°C. In order to develop new disc superalloys allowing an increase in the temperature capability a better understanding of the relative role of creep, fatigue and oxidation on crack growth processes is necessary. The objective is to identify some pertinent criteria which will permit to select appropriate composition and microstructure.

In this work the behaviour under creep-fatigue loading conditions of a P/M Astroloy with the composition Ni-16.4 Co-14.7 Cr-3.9 Al-3.5 Ti-5.0 Mo-0.03 C-0.02 B-0.03 Zr (weight per cent), was investigated at 650°C and 750°C. Crack propagation tests were conducted on SEN 60x13x5 mm³ samples using a 10s - 300s - 10s trapezoidal cycle (300 seconds dwell time at the maximum tensile level) with a load ratio $R = 0.05$. Crack length was determined by a D.C. potential drop technique and all the specimens were precracked by fatigue in air at 650°C with a frequency of 20 Hz. In order to separate the intrinsic properties of the material and the oxidation effects, the creep fatigue experiments were conducted in air and in a high vacuum (pressure $\approx 2 \times 10^{-4}$ Pa).

TEMPERATURE AND ENVIRONMENT EFFECTS

The role of temperature and environment was investigated on a material processed with an argon atomized powder compacted by extrusion. This material has a grain size of 20-30 μm after supersolvus heat treatment.

Role of temperature

The $da/dN-\Delta K$ curves obtained in vacuum are reported in Fig.1. The increase in temperature from 650°C to 750°C has a significant effect on the crack growth rates (about 8 times higher at 750°C than at 650°C)

and is accompanied by a modification of the fracture processes from transgranular at 650°C to intergranular at 750°C ; that shows the importance of time-dependent damage processes at 750°C.

Environmental effects

Fig. 2 illustrates the da/dN - ΔK relationships obtained in air and in vacuum at 650°C. The environment produces a strong acceleration of crack propagation rates especially for the low ΔK values (10 times higher in air than in vacuum) associated with a marked modification of the fracture mode which becomes clearly intergranular in air [1].

At 750°C the effect of environment is more complex (Fig. 3) ; the da/dN - ΔK relationship in air can be represented by two different curves according to the initial ΔK conditions :

- when test in air is started at $21 \text{ MPa}\sqrt{\text{m}}$ the crack growth rate is about 50 times higher in air than in vacuum
- when test in air is started at low ΔK values ($13 \text{ MPa}\sqrt{\text{m}}$) high initial crack growth rates are also observed but da/dN decreases, reaching values identical to that obtained in vacuum for $\Delta K > 20 \text{ MPa}\sqrt{\text{m}}$.

The crack path is intergranular with no significant changes between the two test conditions.

This behaviour in air at 750°C is still not clearly understood. One possible mechanism to explain this striking slowing down of the crack growth rate in air could be the progressive formation for low ΔK of a protective oxide layer limiting the penetration of oxygen at grain boundaries. According to different authors [2,3] , this protective effect could be obtained with the formation of Cr_2O_3 oxides.

EFFECT OF MICROSTRUCTURE AT 750°C.

The importance of creep damage processes at 750°C led us to investigate the role of grain size and grain boundary morphology at this temperature. For this study an Astroloy elaborated with rotating electrode powders compacted by extrusion was used. Three different microstructures have been tested in air and in vacuum : a fine-grained material (20-30 μm) with smooth grain boundaries, a coarse-grained material (65-90 μm) with smooth grain boundaries and a coarse-grained material (45-65 μm) with serrated boundaries ; procedures used to elaborate such materials are detailed in [4].

Effect of grain size

Fig. 4 gives the results obtained in air and in vacuum for the fine and coarse-grained material with smooth grain boundaries. These results show that in air the decrease of the grain size leads to a marked detrimental effect on the creep-fatigue resistance (da/dN near 20 times higher for the fine-grained structure).

At a first sight such results appear to be in good accordance with the role of grain size on creep properties. However this point is not confirmed by the results obtained in vacuum since in this environment the fine grained material exhibits a better resistance than the coarse material. That means that the effect of grain size on crack growth under creep-fatigue loading conditions cannot be simply related to creep properties and that the detrimental role of a fine grained structure observed in air at 750°C is above all associated with environmental effects as it has been noted previously at 650°C [5].

It must be noted concerning the results in air given in Fig. 4 that for the coarse-grained structure the da/dN - ΔK relationship becomes identical to that obtained in vacuum after the transient period observed at low ΔK values. This behaviour is not observed for the fine-grained structure even if in this case the initial ΔK level imposed for starting the creep-fatigue test was lower ; that shows the higher sensitivity of this structure to the environment.

Effect of grain boundary morphology

The results obtained in air and in vacuum on the coarse-grained material with smooth and serrated boundaries are plotted in Fig.5. For the testing conditions used here, results in air and in vacuum are identical after the initial transient period in air. Therefore for these structure only the intrinsic properties of the material in the absence of environmental effects can be discussed from Fig. 5. It can be seen that serrated grain boundaries strongly improve crack growth resistance under creep-fatigue at 750°C. This

effect is explained by a modification of the propagation processes which are clearly intergranular in air or in vacuum for the material with smooth boundaries and which become mixed (intergranular and transgranular) with serrated boundary structure.

CONCLUSION

The rates of crack growth under creep fatigue were measured in an Astroloy tested at 650°C and 750°C in air and in a high vacuum. Tests under vacuum show that the increase in temperature from 650°C to 750°C accelerates crack growth by favouring creep damage ; moreover the fracture path was observed to become intergranular at 750°C while it is transgranular at 650°C.

Environmental effects are already considerable at 650°C, crack propagation rates being about one order higher in air than in vacuum. At 750°C the environmental effects are still stronger (a factor 50 between crack growth rates in air and in vacuum). However at this temperature we have shown that oxidation effects can be suppressed to obtain the same da/dN - ΔK relationship as in vacuum. This suppression of environmental effects, which can only be obtained by starting the creep-fatigue test at low ΔK levels is accompanied by an atypical da/dN - ΔK curve with the crack growth rate decreasing by a factor of about 100 when ΔK increases from 13 to 21 MPa $\sqrt{m}$; This behaviour is assumed to be associated with the formation of a protective oxide layer.

At 750°C, in air, coarse-grained material has a better resistance than a fine-grained material, but the contrary is observed in vacuum showing the importance of microstructure and environment interactions. Serrated grain boundaries can strongly improve the resistance to crack propagation by changing the fracture processes from intergranular to a mixed propagation mode.

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Fig. 1 - Effect of temperature on creep-fatigue crack propagation in vacuum.

Fig. 2 - Creep-fatigue crack propagation data in air and in vacuum at 650°C.

Fig. 3 - Creep-fatigue crack propagation data in air and in vacuum at 750°C for different initial ΔK_i levels. a) vacuum, $\Delta K_i = 21 \text{ MPa}\sqrt{\text{m}}$; b) air, $\Delta K_i = 13 \text{ MPa}\sqrt{\text{m}}$; c) air, $\Delta K_i = 21 \text{ MPa}\sqrt{\text{m}}$

Fig. 4 - Effect of grain size on creep-fatigue crack propagation data in air and in vacuum at 750°C.

Fig. 5 - Effect of grain boundary morphology on creep-fatigue crack propagation data in air and in vacuum at 750°C.