EFFECT OF OXYGEN ON CYCLIC CREEP

FOR ALLOY 718

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Abstract

The time-dependent fatigue and its micromechanisms of superalloy GH169 at 650°C were studied. Under the same stress levels and hold times, the cyclic creep lives in low vacuum (10^{-2} torr) and in high purity Ar (99.99%) were shorter 2 to 5 times than in air and high vacuum (10^{-7}). The damage mechanisms in the former two environmental tests were mainly oxidation-fatigue interactions and their fracture surfaces were revealed to be covered with uncontinuous oxide particles. But the latter two damage mechanisms were creep-fatigue interaction (in high vacuum 10^{-7} torr) and creep-fatigue-oxidation interaction respectively. The fracture surface in air was covered with dense and even oxide films but in high vacuum (10^{-7}), no remarkable oxide trace was observed. As the time prolonged the Cr_2O_3 content in the surface oxides increased in air and decreased in Ar.

Introduction

Time-dependent fatigue which is also termed creep-fatigue or creep-fatigue-environment interactions is the most common damage and failure factor of high-temperature structures. Surface fatigue cracks often originate as intergranular cracks in air at high temperatures, especially at low frequencies. Both environment (particularly oxidation) and time-dependent flow can influence the propensity for intergranular surface crack initiation, since cracks are often developed as a consequence of the grain boundary sliding, void coalescence, and grain boundary oxidation [1]. Till now, many authors focused at determining the stress, temperature, gas chemistry and metallurgical effects on time-dependent fatigue and among them the fatigue life research of strainless steels, aluminium alloys and nickel-base alloys in high temperature vacuum and air by Coffin [2] and Mar-M200 single crystal superalloy by Antolovich [3] were the representatives. While other studies [4] were basic investigations of environmental damage mechanisms and models as that of oxidation effects by Floreen and Raj [5]. Guo Jianting [6] compared the creep-fatigue-environment interaction of IN738LC superalloy in vacuum, air and hot salt aggressive environment. In recent years, Liu et al [7] suggested two primary damage mechanisms of high-temperature low cycle fatigue-creep and oxidation. Both of these two mechanisms are thermally activated. In the present paper, the high-temperature oxidation effects on time-dependent fatigue and its mechanisms of GH169 are studied.

Materials and Experimental Analysis

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Superalloy GH169, similar to alloy IN-718 and used commonly as turbine disk, is a nickel superalloy precipitate-strengthened with b.c.c. $\text{Ni}_3\text{Nb}(\gamma'')$ phase and f.c.c. $\text{Ni}_3(\text{Al}, \text{Ti})(\gamma')$ phase. The heat-treatment is: 960°C, 1 h, air cooling; 760°C, 8 h, furnace cooled to 620°C, 8 h air cooling. The chemical composition of it is(wt-%) C 0.047, Mn<0.1, Fe 2.19, S<0.002, P<0.005, Si<0.16, Cr 19.20, Al 0.53, Ti 1.10, Pb 0.0007, Bi 0.0005, B 0.00005, Cu<0.069, Co<0.10, Mg<0.0012, Ca<0.01, Nb 5.09, Me 2.86, Ni residual. The mechanical properties of it is shown in Table 1.

Table 1 Mechanical properties of GH169

Temp. °C	σ_b MPa	$\sigma_{0.2}$ MPa	δ %	ψ %
RT	1402	1224	21	42
650°C	1120	981	18	—

Tests were carried out on a modified MP-3GA cyclic creep test machine at 650°C in air, and the specimens were the standard low cycle fatigue ones. The loading was stress-controlled, rectangular wave and the hold time of peak value was 1 or 10 min.. The loading and unloading time were 17 sec.. The environmental tests were firstly to creat a vacuum 0.1 Pa and then to pass through high purity Ar(higher than one atmosphere pressure. Other environmental tests were in vacuum conditions from high vacuum(10^{-7} torr) to low one(10^{-2} torr) which were created by a high temperature vacuum furnace composed of mainly a supper high vacuum turbine molecular pump(10^{-9} torr) and a rather small vacuum chamber(tubal shape) heated with an electric-resistance wire furnace around the tube.

The monography of fracture surfaces for some specimens was observed firstly with scanning electronic microscope, then surface oxidation films were cleaned with NaOH and KMnO_4 solution to study the environmental effect. Finally, the surface constituents of the fracture were analyzed with X-ray photoelectron spectroscopy(XPS).

Data analysis of the environmental test Table 2 indicated the cyclic creep fracture test data of GH169 at 650°C in different environments.

Table 2 Cyclic creep lives for GH169 at 650°C in various environments

No. of spec.	Stress (MPa)	Hold time (Min.)	Vacuum deg. (Torr)	Lives(cycles)
K(2)	785	10	10^{-7}	204
K(8)	785	10	Air	194
K14	785	10	5×10^{-4}	193
K13	785	10	10^{-2}	130
K2	785	10	Ar	43
K20	785	1	10^{-7}	1140
Kd	785	1	Air	1021
K(2)	785	1	Ar	398
K5	735	10	10^{-7}	334
K(5)	735	10	Air	438
K2	735	10	Ar	82
K24	735	1	10^{-7}	2000
K25	735	1	Air	1562
K18	735	1	10^{-2}	960
L24	785	2	10^{-7}	1400 min.
L17	785	2	Air	960 min.
L21	785	2	Ar	720 min.

The ratios of the cyclic creep lives on the same stress levels and hold times but in different environments were as follows;

on 785 MPa, 10 min. hold time condition,

$$N(10^{-7}); N(10^{-2}); N(\text{Air}) = 5:1:5$$

on 785 MPa, 1 min. hold time condition,

$$N(10^{-7}); N(10^{-2}); N(\text{Air}) = 3:1:3$$

on 735 MPa, 10 min. hold time condition,

$$N(10^{-7}); N(10^{-2}); N(\text{Air}) = 4:1:5$$

on 735 MPa, 1 min. hold time condition,

$$N(10^{-7}); N(10^{-2}); N(\text{Air}) = 2:1:1.7$$

Figure 1 presented the effects of oxygen on cyclic creep lives of GH169 at 650°C in different atmosphere pressures from (10^{-7} torr), Ar to air pressure. The most remarkable cyclic creep damage were nearly in the neighbor of atmosphere pressure 10^{-2} torr and Ar in which a low valley of lives is envisaged on the same stress level and same hold time.

Fig.1 The cyclic creep lives of GH169 at 650°C in different atmosphere pressures

Fig.2 The hold times effects on the cyclic creep lives in different environments

In high vacuum (10^{-7} torr) or no oxygen environment, the cyclic creep life is that of true creep-fatigue interaction while the life in air or the full of oxygen content is that of creep-fatigue-oxidation interactions. Both lives in above two environmental

conditions approximate each other because a dense oxide film formed by oxygen in air on the surface of the specimen tested protects it from further aggression with oxygen. But the cyclic creep lives in low vacuum(10^{-2}) or Ar are 2-5 times less than those in high vacuum or in air. In above different environments the cyclic creep lives may reduce 3-4 times more or less from hold time 1' to 10' under same stress level shown as figure 2.

Micrographic observation, SEM observation showed that scattered oxide inclusions have been found on the surface and at the grain boundaries in the specimens tested in Ar (fig.3c). With increasing hold time, the inclusions increase, but no dense oxide film could be seen(Fig.3d). On the contrary, a dense oxide film covered on the fracture surface of the specimens tested in air and became thicker and thicker with the increase of holding time(Fig. 3a,3b).

a. 1 min. in air 1500X

b. 10 min. in air 1500X

c. 1 min. in Ar 2000X

d. 10 min. in Ar 2000X

Fig.3 Fracture surface of GH169 at 650°C in Ar and air

Fig. 4 XPS spectra of element Cr for GH169 in Ar (a),(b), Air (c),(d)
(a),(c) 1 min. ; (b),(d) 10 min.

Fig.4 shows the results of XPS analyses for the fracture surfaces of the specimens tested in Ar and air. It indicates that the Cr_2O_3 particles in specimens tested in Ar decrease with holding time(Fig.4a,4b), the nonsequence of oxide film causes the more severe oxidation on local region of the surface. On the other hand, in air the Cr_2O_3 content in oxide film increases(Fig.4c,4d), and thus the density of Cr_2O_3 prevents the surface from diffusing of oxygen into the interior and delays the internal oxidation. Secondly, no matter on crack initiation and propagation zones the micrographic structures of the fracture have the transition from transgranular to intergranular accompanied with high vacuum(10^{-7} torr) to low vacuum(10^{-2} torr) and Ar tests shown as Fig.5,6 and 7. The latter two present the increasing of intergranular cracks that is the strengthening of grain boundary oxidation. This may be used to explain the low valley phenomena appeared in the neighbor of low vacuum(10^{-2} torr) and in the high purity Ar.

Fig.5 K2 (10^{-7} , 10') 500X

Fig.5,6,7
The micro mechanisms of the cyclic
creep fracture of GH169 at 650°C in

10^{-7} torr (Fig.5)
 10^{-2} torr (Fig.6)
Ar (Fig.7)

Fig.6 K16 (10^{-2} , 1') 360X

Fig.7 K(2) (Ar, 1') 360X

Fig. 8a K20 (10^{-7} , 1') 1000X

Fig. 8b K18 (10^{-2} , 1') 1000X

Fig. 8c (Ar, 1') 1000X

Fig. 8d (Air, 1') 1000X

Fig. 8 The Oxygen content effects of GH169 at 650°C
in different vacuum environments, Ar and Air

Discussion

The effects of Oxygen on cyclic creep lives of GH169 at 650°C in different vacuum environmental tests and in high purity Ar compared with air were illustrated as Fig.8. In high vacuum(10^{-7} torr) the grain boundary and the dimples within the grain are clean and no remarkable oxidation trace shown in Fig.8, while in air, as Fig.8d, a dense oxides protective film covered on the fracture surface prevents further grain boundary oxidation. In low vacuum(10^{-2} torr) test, there exists a little of carbides, as shown in Fig.8b, and in Ar as Fig.8c, the uncontinuous oxides particles can play the detrimental effects on the cyclic creep lives. So do the fracture surface of pure creep specimen in above various environmental tests.

The above environmental tests indicate the extensive evidence that air is a very aggressive environment and that oxygen is the element particularly responsible. Even very minor amounts of oxygen can accelerate fatigue-growth rates comparable to those produced by air. In some cases minor amounts of oxygen may be more damaging than higher levels[8]. In our above tests the formed low valley of cyclic creep lives in low vacuum (10^{-2} torr) and Ar indicates that the environment for lack of oxygen may be the more important factor than creep to fatigue and its life. The studying of the minor amounts of oxygen on the lives for GH169 has the practical meaning, in general the majority portion of Oxygen is used to assist combustion and the residual portion of it between the interface of the high temperature structures and the gas is much more less than it in air which accelerates the damage of the components because of the main oxidation-fatigue interaction.

Mechanisms of time-dependent fatigue damage in Ar and air. Raj[9]proved through microstructural observation that oxygen can diffuse along the grain boundaries from the environment to the structure interior. The character of this environmental sub-surface damage is the formation of microcracks or cavities along the grain boundaries at the front of crack tip. Oxygen diffuses along grain boundaries and reacts with second-phase precipitates to produce a very thin layer of oxide, then leads to cavitation. Under the internal high pressure, γ' -phase particles of Ni-base superalloys may be easily oxidized and loses their strengthening effect. The formation of protective layer of chromium oxide and nickel oxide makes the different activities between the oxygen in the environment and that beneath the oxide scale. The deteriorious effect of oxygen is likely only if the oxide scale is broken and fresh surface is exposed. In the environmental test of GH169, the paucity oxygen in Ar and the residual oxygen in the vacuum container can react with the elements which are easily oxidized at first. While it is the turn of chromium to react with oxygen, there exists no enough oxygen to do with it. This results in no fine and even Cr_2O_3 protective layer covered on the fracture surface and only be discontinuous Cr_2O_3 particles form, this makes the deteriorious effect of oxygen continue. On the contrary, the active effect of oxygen in air would be prevented from the dense and even Cr_2O_3 protective layer due to the enough oxygen. SEM and XPS experimental evidence support this idea and so did Choe et al[10].

Conclusion

- 1) The time-dependent fatigue lives of superalloy GH169 at 650°C in various environments decreased significantly with increasing hold time, and the fatigue fractures were mainly intergranular. Under the conditions of the same stress levels and duration, the cyclic creep lives in low vacuum(10^{-2} torr) and in high purity Ar(99.99%) were shorter 2 to 5 times than those in air and high vacuum(10^{-7}).
- 2) The cyclic creep damage mechanisms for GH169 at 650°C were dependent on different environmental conditions, in high vacuum(10^{-7} torr), its may be mainly creep-fatigue interactions and in low vacuum(10^{-2} torr) and in Ar principally oxidation-fatigue one. But in air or full of Oxygen, the damage mechanism of GH169 at 650°C may be creep-fatigue-oxidation interactions.
- 3) In high temperature atmosphere, the fracture surface of GH169 was revealed to be

covered with a dense and even oxide film among which the Cr_2O_3 content increased as the time prolonged but in Ar, with an discontinuous oxide particles in which Cr_2O_3 content decreased with the time.

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