THE EFFECT OF PARTIAL VACUUM ON THE FATIGUE CRACK GROWTH OF NICKEL BASE SUPERALLOYS

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Abstract

The effect of a partial vacuum environment, simulative of a typical spin-pit test atmosphere, on the high temperature crack growth behavior of two nickel-base superalloys was examined. Under constant amplitude loading, the partial vacuum typically reduced the crack propagation rate in one alloy while an increase in growth rate was found for the other. Cycling with a superposed tensile dwell period increased the crack growth rates at low levels of ΔK relative to those found in air for both alloys. This anomalous behavior was heretofore not recognized and raises doubt as to our understanding of the environmental drivers on fatigue crack propagation.

Introduction

There is a growing interest to improve the durability of superalloy gas turbine engine components and a desire to understand their suitability for long hot time applications. Of particular interest is an understanding of their performance in long hold time applications such as sustained supersonic flight and/or industrial gas turbine application. These applications will require a better characterization of the influences of creep and environment on their mechanical behavior. With the Air Force's requirement for damage tolerance, an assessment of the crack growth behavior of these materials is required to assess their suitability for these new missions. Currently there is no generally accepted understanding of the mechanism(s) by which crack growth is influenced by environment [1-7]. The primary environmental driver affecting crack growth is thought to be oxidation [1, 2], however, moisture appears to have some influence on the high temperature crack growth [3] or, more likely, a combination of both moisture and oxidation is important. Often gas turbine engine components are spin tested to validate their design methodology and to assess their damage tolerance to small flaws, e.g. [8]. These spin tests are typically conducted in pits evacuated to a moderate vacuum level to minimize the aerodynamic heating (and reduce the power consumption) of bladed disks as well as satisfy health and safety requirements. However, the effect of the partial vacuum is seldom taken into account in the assessment of the design methodology, and there is a dearth of experimental data regarding the influence of this environment.

Experimental Program

Tests were conducted at two intermediate vacuum levels to assess the influence of a spin pit environment on the crack growth behavior of several nickel base superalloys. Only results for powder metallurgy IN-100 and conventionally wrought Waspaloy will be reported here as these represent common materials in use

today. (All Ni-base superalloys examined to date exhibit, qualitatively, similar behavior.) Compact tension specimens were obtained from forgings of these materials and were tested in a servohydraulic fatigue rig fitted with a test chamber capable of an ultimate vacuum of 10^{-7} torr. The partial vacuum conditions were maintained using a rotary vane pump (only) throttled to reduce its suction and the vacuum levels were maintained using a precision leak valve. This dynamic atmosphere is thought to accurately reproduce that found in a spin pit with the potential exception of gaseous species such as carbon that could result from dissociation of hydrocarbons present in the turbine lubrication system. The intermediate vacuum levels of 100 and 450 mTorr were examined which roughly represents the range of vacuum levels incorporated in spin pit tests. The partial vacuum was controlled to better than 20 mTorr. The specimens were heated to the test temperature using a standard resistance furnace inside the atmospheric chamber. Identical tests were conducted in laboratory air on a second test machine to determine the baseline crack growth resistance. The crack lengths were measured using the direct current potential difference method and verified with measurements on the fracture surfaces. All tests were conducted using a K controlled procedure whereby blocks of constant loading condition were applied to the specimen for an interval of growth, 0.4 to 0.6 mm, in order to attain a constant growth rate. The maximum K levels were always maintained constant or increased from one block of loading to the next to minimize load interaction effects. Overall, this technique allowed a rapid interrogation of the material's behavior under a wide range of loading conditions and minimized specimen-to-specimen variability in the results.

Results

IN-100

IN-100 was tested at temperatures of 621° and 657°C using a sinusoidal waveform having a 30 second cyclic period - with and without a hold time at maximum load of 60 seconds. The stress ratio (R) was 0.3 at 621°C and 0.15 at 657°C. Tests were conducted at 1 atmosphere, 100 mTorr, 450 mTorr, and 10^{-5} torr to assess the environmental effects on the above loading conditions. Crack growth rate as a function of ΔK is shown in Fig. 1 for the constant cycling (no hold): a) 621°C tests and b) 657°C tests. Here, as expected, the crack growth rates layer with atmospheric content: the highest concentration of air has the highest crack growth rate and the lowest concentration of air (10^{-5} torr) has the lowest. It is readily apparent that the crack growth rate at partial vacuum is reduced from that in air and is influenced by the relatively modest change in partial pressure from 450 to 100 mTorr, which is in the range where most spin tests are conducted. This is especially apparent at the higher temperature at the lower crack growth rates, Fig 1(b). The crack growth

behavior for the 30 second cycle with a 60 second hold time at maximum load is shown in figure 2 for: a) 621°C and b) 657°C. Here it is surprising to find that at low levels of ΔK the crack growth rates at the poor vacuum levels are equal to or greater than those found under ambient (1 atm) conditions. At higher ΔK levels, the growth rates follow the normal trend that was found under the constant cycling conditions. The flatness of the crack growth rate curves under the partial vacuum condition, i.e. growth rates somewhat independent of ΔK , is more representative of an

environmentally driven crack growth mechanism. Similar to the finding under constant amplitude cycling, the sensitivity to the partial vacuum level is more pronounced at the elevated temperature. Also note in both cases that the difference in crack growth rates between 1 atm and 10^{-5} torr increases slightly as the temperature is increased from 621 to 657°C – a small 5% increase in temperature.

Figure 1. Crack growth rate in IN-100 versus applied ΔK for a 30 second cycle with; a) 621°C, $R=0.3$, and b) 657°C, $R=0.15$

Figure 2. Crack growth rate in IN-100 versus applied ΔK for a 30 second cycle with a 60 second hold at maximum load; a) 621°C, $R=0.3$, and b) 657°C, $R=0.15$.

Figure 3. Crack growth rate in Waspaloy versus applied ΔK for a 60 second cycle at 650°C, $R=0.25$ for; a) constant cycling and b) with a 60 second hold time at maximum load.

Waspaloy

Waspaloy was tested at only one temperature, 650°C, with a 60 second sinusoidal period and $R = 0.25$ - with and without a 60 second hold time. Pressures of 1 atm, 100 mTorr and 450 mTorr were interrogated. The crack growth rates for constant cycling and cycling with a hold are shown in figure 3a and b, respectively. Here, even under constant cycling at low ΔK 's, the crack growth rate at 450 mTorr is faster than 1 atm. Under the cyclic condition with the 60 second hold time, the crack growth rates at both partial vacuum levels is greater than the 1 atm condition – even at ΔK levels as high as 30 MPa√m. Under the partial vacuum of 450 mTorr, the increase in crack growth rate is particularly striking!

Crack Growth Transients in IN-100

The test technique incorporated in this study also allowed a look at the crack growth transients when switching from one loading or environmental condition to another. As the loading was constant for each condition examined, a constant crack growth rate was expected. That is, given a temperature, R , and waveform, with constant K conditions, a linear crack length versus cycles trace should result unless a variation in crack growth rate was introduced by the prior loading. In IN-100, a repeatable transient was observed when crack growth conditions changed from the partial vacuum condition to the high vacuum, or vice versa. Figure 4 shows the two types of transient for $K_{max} = 20$ MPa√m, $R=0.15$ in terms of crack increment versus normalized cycles from the start of the new condition. (The cycles had to be normalized due to the large difference in crack growth rate.) The dashed lines approximate the steady state crack growth rates. It was found that when the vacuum was improved from an intermediate value (99 mTorr shown) to 10^{-5} torr, the initial crack growth rate at 10^{-5} torr was depressed. In all cases, the average crack growth rate during the transient was reduced by a factor of 2 to 5 for 0.1 to 0.2 mm independent of the K level or loading condition – the transient existed both with and without a hold time

in the cycle. When the vacuum level was degraded from 10^{-5} torr to an intermediate value, the crack growth rate at the intermediate value was initially accelerated before attaining a constant growth rate. Again, the average crack growth rate during the transient was always increased by a factor of 2 to 4 for 0.1 to 0.2 mm independent of the K level or loading condition. These transients are, for example, inconsistent with an accumulation of damage (e.g. oxide formation) ahead of the crack tip in the partial vacuum as will be discussed later.

Figure 4. Transient crack growth in IN-100 at 657°C, $K_{max} = 20$ MPa√m, $R=0.15$ in terms of crack extension for a given loading condition versus cycles normalized by the total cycles for the given loading condition.

Fractography

The fracture modes under the various test conditions were examined with a field emission scanning electron microscope using secondary electron imaging. Figure 5 shows a typical comparison of the hold time fracture surfaces for IN-100 from crack growth at 450 mTorr and 1 Atm. The fracture surfaces are both intergranular yet there appears to be more out of plane deviation on the specimen tested in the partial vacuum. Furthermore, specimens tested in the partial vacuum environment showed a greater oxide depth as compared to the specimen tested in air. Figure 6 shows the fracture surfaces from Fig. 5 in better detail as well as the 10^{-5} torr fracture surface having the same loading conditions. The oxide on the fracture surface in Fig. 6(c) is due to the time the specimen spent at temperature under partial vacuum conditions. The specimen never saw air exposure at temperature, but there was enough oxygen in the partial vacuum to produce the nodular oxide scale. The underlying fracture is considerably flatter than that obtained at higher pressures and is assumed to be transgranular. (Additionally there was little difference in the fracture behavior at 10^{-5} torr for continuous cycling and cycling with a hold period of 60 s.) The fracture surface at 450 mTorr, fig. 6(b) shows similar nodular oxides as the fracture surface produced at 10^{-5} torr, but also shows a “feathery” oxide that is filling the valleys between the grain boundaries. This oxide is completely absent in the specimen tested in lab air, fig. 6(a) where even the oxide nodules are not as well defined. This could be due, in part, to the longer test time of the specimen tested in partial vacuum due to the very slow growth rates at 10^{-5} torr which at least doubled the total time of exposure. Yet this specimen was never exposed at temperature to an atmosphere greater than approximately 500 mTorr.

In Waspaloy, no obvious differences were observed in the fracture behavior at both partial vacuum levels and laboratory air, see figure 7. In both 1 atm and 450 mTorr environments, the fracture mode is predominantly intergranular, as expected though some transgranular facets are also found. Here no differences in oxide

scale were found. This could be due to the smaller specimen used for this test and the lack of 10^{-5} torr crack growth that substantially reduced the test time.

Discussion

The influence of environment on the crack growth behavior of nickel-base superalloys has been studied by a number of researchers over the years, e.g. [1-7] and the most studied material has been Alloy 718 [1-4, 7]. The current study has extended the environmental understanding to two additional superalloys, IN-100 and Waspaloy, and has broadened the understanding by looking at the temperature and/or K sensitivity. One simple way to look at the environmental sensitivity discovered here is to calculate an environmental factor (EF) – this being the ratio of the crack growth rate under a given atmosphere to the crack growth rate for the same loading conditions in laboratory air. Figure 8 shows the trends calculated from the data in figs. 1-3 for the cycling; (a) without a hold time and (b) with the hold time. Notice that $EF = 1$ indicates no environmental effect while $EF > 1$ indicates a more aggressive environment and $EF < 1$ indicates a more benign environment.

It is apparent that Waspaloy is more aggravated by the partial vacuum environment than IN-100 and that it is more sensitive to small changes in environment – there is a greater separation between the 100 mTorr and 450 mTorr curves. At low K levels, even the constant amplitude cycling produces an increased crack growth rate relative to air. It is clear that IN-100 is more sensitive to environment at low K levels and more sensitive to changes in environment at the higher temperature. The reasons for the different behavior in the two materials is not obvious, nor is the reason behind an increase in crack growth rates at low k levels under a protected environment.

Figure 5. Fracture surfaces of IN-100 with an applied $\Delta K = 14 \text{ MPa}\sqrt{\text{m}}$, $R=0.3$, 621°C , and a 30 second cycle with a 60 second hold time: a) 1 atm and b) 450 mTorr.

Figure 6. Details of fracture surfaces of IN-100 with an applied $\Delta K = 14 \text{ MPa}\sqrt{\text{m}}$, $R=0.3$, 621°C , and a 30 second cycle with a 60 second hold time: a) 1 atm, b) 450 mTorr, and c) 10^{-5} torr.

Figure 7. Fracture surfaces of Waspaloy with an applied $\Delta K \sim 19 \text{ MPa}\sqrt{\text{m}}$, $R=0.25$, 650°C , and a 60 second cycle with a 60 second hold time: a) 1 atm and b) 450 mTorr.

Figure 8. The environmental factor ($EF = \frac{da/dN_{\text{environment}}}{da/dN_{\text{air}}}$) as a function of $K_{\max}$ for IN-100 and Waspaloy; a) continuous cycling and b) with hold times (note the different scales). The complete test conditions are found in figs. 1-3.

Transient Behavior

The transient crack growth behavior in Fig. 4 provides some insight into the damage processes of the intermediate vacuum. If the intermediate vacuum produced a diffuse damage zone ahead of the crack tip, then the opposite trend would be expected, i.e. a *decreasing* growth rate to steady state when switching from poor vacuum to high vacuum as opposed to the observed *increasing* crack growth rate to a steady state. The observed trends however are more likely caused by changes in the fracture mode and differences in crack tip sharpness. A transition region (99 mTorr to 10^{-5} torr) in IN-100 is shown in figure 9. Clearly there is a transition from intergranular to transgranular fracture and the crack front of the intergranular fracture is neither straight nor planar. The transgranular crack growth in 10^{-5} torr is retarded by the out of plane tortuosity of the crack tip that tends to act like a blunted crack. The secondary cracking also associated with the intergranular fracture (see Fig. 5) also would tend to reduce the effective K at the crack tip.

Figure 9. Fracture surface of IN-100 tested at 657°C, $R=0.15$, $K_{max} = 20 \text{ MPa}\sqrt{\text{m}}$ with an environment of 99 mTorr on the right and 10^{-5} torr on the left. (Crack growth direction is right to left.)

The other repeatable transient occurred immediately after resuming crack growth under partial vacuum conditions following 10^{-5} torr cracking. As shown in Fig 10, the crack front during 10^{-5} torr crack growth is very straight and the crack tip is undoubtedly sharp from the transgranular fracture. The sharpness of the crack tip would tend to concentrate the stress at the crack tip and thereby increase the driving force for environmental attack of the crack tip material [2]. The cracking shown in Fig. 10 appears to immediately become intergranular in the poor vacuum condition.

Figure 10. Fracture surface of IN-100 tested at 657°C, $R=0.15$, $K_{max} = 20 \text{ MPa}\sqrt{\text{m}}$, 60 s hold at maximum with an environment of 10^{-5} torr on the right and 99 mTorr on the left. (Crack growth direction is right to left.)

The partial vacuum conditions of the current study are 1 atm, 100 mTorr, 450 mTorr, and 10^{-5} torr and can be estimated to have oxygen partial pressures of 159 torr, 9×10^{-2} torr, 2×10^{-2} torr and 2×10^{-6} torr, respectively – assuming oxygen is 20.95% of the atmosphere and is equally pumped by the vacuum pumps. Using this assumption, the present results can be compared to the earlier work. Andrieu, and co-workers, [2,7] have examined the influence of oxygen partial pressure on the fracture and crack growth behavior of Alloy 718 under 650°C dwell fatigue conditions and did not find an increase in crack growth rate over that found in laboratory air. A steady decrease in crack growth rate was found for the 10-300-10 cycle with a K of 20 $\text{MPa}\sqrt{\text{m}}$ over the oxygen partial pressure range from 7.5×10^{-2} to 7.5×10^{-4} torr. The upper end of this range is exactly the pressure where the increase in growth rate has been found in the present study. Bache, et al., [4] examined the influence of a 150 Pa “spin rig” environment (~ 0.2 torr oxygen partial pressure) on the dwell (30 second) crack growth in Alloy 718 at 600°C. Surface crack specimens were used in this study and only a *decrease* in crack growth rate in the surface direction was found relative to the depth at this partial pressure. In Alloy 718, there is a documented influence of oxygen partial pressure on the oxide formation [1-3,7]. Under atmospheric conditions down to an oxygen partial pressure of approximately 10^{-4} torr a non-protective, spinel oxide forms of the NiFeO type [2,7]. This is the expected oxidation behavior for all of the partial vacuum tests conducted here. That is, a protective Cr_2O_3 oxide layer is not expected to form at the intermediate vacuum levels of the present study. The expectation therefore would be that the crack growth rates should be relatively unchanged from those in laboratory air. Browning, et al., [3], however, have found that in a $\text{H}_2/\text{H}_2\text{O}/\text{inert}$ gas environment that rapid crack growth behavior in Alloy 718 can exist with an oxygen partial pressure as low as 10^{-23} torr in a high moisture environment. They concluded that a better knowledge of the partial pressures of both oxygen and water vapor was necessary to

predict a material's environmental sensitivity. Clearly the 20 orders of magnitude difference in oxygen partial pressure between the two researcher groups must be resolved.

The puzzling behavior found in two alloys in this study is not fully understood at this time. It may be due to changes in protective oxide scale formation or differential pumping rates. Careful work is necessary to understand the complex chemical and mechanical interactions at a growing crack tip.

Impact

The present findings are certainly puzzling and mechanism(s) of the anomalous increase in crack growth rates in a partial vacuum are unknown. However, the impact of these findings are documented by Russ, et al. [9] with respect to a spin test conducted to assess the suitability of current life prediction methodologies to predict end of life of a cracked test article subject to a spin pit loading. Without the incorporation of advanced, 3-D fracture mechanics, processing induced residual stresses *and* these anomalous cracking results, the test article life could not have been predicted accurately. Few gas turbine engines operate in a partial vacuum, so the direct impact of the present findings on aircraft operation are limited, but the accuracy of life methods that have been "validated" by high temperature component spins [8] may be in question.

Finally, the present results cause one to question the behavior of materials under actual gas turbine engine operating conditions where the effects of high pressure air in the compressor and combustion products in the turbine are rarely taken into account in the lifing methodology. With the desire for sustained supersonic flight, clearly superalloy research should address this question as we continue to ask more and more from this class of materials!

Conclusions

Tests were conducted on two nickel-base superalloys to assess the influence of a partial vacuum level on the fatigue crack growth rate. Under the time dependent loading conditions examined a reduction in crack growth rate was observed typically observed in IN-100 for continuous cycling while an *anomalous* increase in fatigue crack growth rate was apparent at low K levels for cycling including a hold period at maximum load when compared to partial vacuum crack growth rates to those observed in laboratory air. Under the same environmental conditions, Waspaloy exhibited an increase in crack growth rate even in the absence of a hold period at maximum load. The causes(s) of this behavior is not understood but clearly points to the necessity for further research in this area.

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References

1. Pineau, A., 1996, "Fatigue and Creep-Fatigue Behavior of Ni-base Superalloys: Microstructural and Environmental Effects," in Mechanical Behavior of Materials at High Temperature, C. Moura Branco, et al., Eds., Kluwer, Netherlands, pp. 135-154.
2. Molins, R., Hochstetter, G., Chassaigne, J.C., and Andrieu, E., 1997, "Oxidation Effects on the Fatigue Crack Growth Behavior of Alloy 718 at High Temperature," *Acta Mater*, 45(2) pp. 663-674.
3. Browning, P.F., and Henry, M.F., 1977, "Oxidation Mechanisms in Relation to High Temperature Crack Propagation Properties of Alloy 718 in H₂/H₂O/Inert Gas Environments," in Superalloys 718, 625, 706 and Various Derivatives, E.A. Loria, Ed., TMS, pp. 665-678.
4. Bache, M.R., Evans, W.J., and Hardy, M.C., 1999, "The Effects of Environment and Loading Waveform on the Fatigue Crack Growth in Inconel 718," *Int. J. Fatigue*, 21, pp. S69-S77.
5. Tong, J., Dalby, S., Bryne, J., Henderson, M.B., and Hardy, M.C., 2001, "Creep, Fatigue and Oxidation in Crack Growth in Advanced Nickel Base Superalloys," *Int. J. Fatigue*, 23, pp. 897-902.
6. Knowles, D.M., and Skelton, D.K., 2001, "High Temperature Fatigue of a Polycrystalline Nickel Base Superalloy," *Materials Science & Technology*, 17(11), pp. 1403-1412.
7. Andrieu, E., Cozar, R., and Pineau, A., 1988, "Effect of Environment and Microstructure on the High Temperature Behavior of Alloy 718," in Superalloy 718 – Metallurgy & Applications, E.A. Loria, Ed., TMS, pp. 241-256.
8. Mills, M.S., DeLuca, D.P., Cowles, B.A., Harris, J.A., Jr., "Engine Retirement for Cause: Volume III – Methodology Demonstration & Validation," AFWAL-TR-87-4069, Wright-Patterson AFB, OH, 1987.
9. Russ, S.M., et al., "Demonstration of advanced life-prediction and state-awareness technologies necessary for prognosis of turbine engine disks," to be published in Health Monitoring and Smart Nondestructive Evaluation of Structural and Biological Systems III, 2004.