# CRACK GROWTH BEHAVIOUR OF NICKEL-BASE HIGH TEMPERATURE ALLOYS AT 500 TO 1000°C

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# **INTRODUCTION**

Efficient and environmentally benign production of electrical power in fossil fired industrial gas turbines, either in single cycle or in combined cycle (gas turbine - steam turbine) plants will require components made of Nibase alloys. Although such materials were developed and are extensively used in aero engines, the much larger dimensions of the components in stationary gas turbines mean that the impact of the much larger scale fabrication on the microstructure and the flaw tolerance of the materials must be investigated. Furthermore, the load cycles are very different; in aero gas turbines, full power operation is only required at take-off and landing, whereas industrial turbines must operate at full power for prolonged periods of time.

Only limited information exists in the open literature regarding the behaviour of technical cracks and the controlling mechanisms of crack growth in Ni-base alloys. Safe operation and estimation of the reliability and the allowable types and numbers of operational cycles require knowledge concerning crack growth initiation resulting from the local inhomogeneities expected in each component, and concerning the growth behaviour under operational loadings, especially creep and creep-fatigue cycles. The impact of the working environment on deformation behaviour is of specific importance. Three candidate alloys for the turbine rotors and discs , and one alloy for the turbine blading were investigated:

- solid solution hardened INCONEL 617;
- $\gamma'$  hardened alloy Waspaloy;
- INCONEL 706, precipitation hardened by  $\gamma'$ ,  $\gamma''$  and  $\eta$ ;
- single crystalline CMSX-4, hardened by a large volume fraction of  $\gamma'$  precipitates.

# **EXPERIMENTAL DETAILS**

#### Materials

The nominal composition and the microstructures of the four test materials are shown in Figure 1. The alloy INCONEL 617 is a Ni-Cr alloy solid solution strengthened by additions of Co and Mo and was originally developed as a sheet material for aero gas turbine combustion chambers. This alloy is a typical example of a forgeable material used in the solution heat treated condition. The material INCONEL 706 is a Nb containing Ni-Fe-base alloy with good forgeability for applications for large scale components. This alloy is strengthened by a complex structure of  $\gamma'$ ,  $\gamma''$  and, dependent on the heat treatment,  $\eta$  phase precipitates. Waspaloy is a  $\gamma'$  hardened material with a low C content leading to a small amount on M<sub>23</sub>C<sub>6</sub> precipitates on the grain boundaries. Because of the high Ti/Al ratio  $\gamma'$  precipitates in a bimodal size distribution of primary and secondary  $\gamma'$  particles. Both alloys may be candidates for applications as rotors or disks in steam turbines with very high steam temperatures of about 700 °C. Single crystalline superalloys, first developed for aero gas turbine blades, exhibit a significant improvement in creep and fatigue resistance over conventionally cast, equiaxed superalloys, allowing about 80C higher materials temperature in operation. Alloy CMSX-4 is a typical second generation, single crystal material with about 70 vol%  $\gamma'$  and solid solution strengthening of the  $\gamma$  matrix by 3 wt.% Re.

alloy	nominal chemical composition in mass-%											hardening
	Ni	Cr	Fe	Co	Мо	AI	Ti	Nb	Та	W	С	
IN 617	bal	23	<2	12	9	1	0.5	2.9	-	-	0.05	sol.hard.,carbides
Waspaloy	bal.	19	0.5	14	4.5	1.2	3.1	0.01	-	-	0.03	γ' phase
IN 706	bal.	16	37	-	-	1	1.5	3	-	-	0.01	γ', γ", η phases
CMSX-4	bal	6.4	-	9.6	0.6	5.6	1	-	6.5	6.4	-	+3% Re,γ' phase



Figure 1: Nominal chemical compositions and microstructures of test materials

#### Test methods

12.7 mm CT specimens (ASME standards E 399 and E647) were machined from the wrought materials with guide notches if necessary (10% of the thickness of specimen, 60° angle). Specimens were fatigue pre-cracked at room temperature up to a depth-to-width ratio of 0.3 - 0.4. Assuming linear elastic fracture conditions [1,2], the results were interpreted using the stress intensity factor,  $K_I$ 

$$K_{I} = \frac{F_{max}}{B \cdot W^{1/2}} \cdot f(a/w)$$
(1)

where  $F_{\text{max}}$  = maximum of applied force, *B*, *W* = dimensions of the specimen, *a* = crack length and f(a/w) a geometrical factor.

INCONEL 617, Waspaloy and INCONEL 706 tested at 700°C or higher do not behave linear elastically, but viscoplastically. If the opening velocity  $\dot{V}$  of the crack is considered, the integral of deformability (C\*) is obtained by

$$C^* = \eta \cdot \sigma_{net} \cdot \overset{\bullet}{V} \tag{2}$$

For CMSX-4, single edge notched (SEN) specimens were manufactured from <001> orientated cast plates. The K<sub>I</sub> function for SEN specimens under stress load is normally given by

$$K_I = \sigma \ \sqrt{\pi a \cdot c} \tag{4}$$

Specimens with corner cracks exhibit a much more complicated stress distribution ahead of the crack tip. This specimen represents the realistic crack geometry within a component due its three dimensionality. Mathematical results are given in [3,5,6], whereby a square edge crack surface area is estimated. One differentiates between the stress intensity factors along the surface of the specimen and in the direction of 45°. Then the stress intensity factor across the whole crack surface may be estimated by

$$K_{I mean} = \frac{K_{I 45^{\circ}} + K_{I surface}}{2} = \left(0.97 - 0.09 \left(\frac{a}{w}\right)^2\right) \cdot K_{I surface}$$
(5)

The approximations help in understanding the crack propagation of a corner edge crack [4]. The experimental observations indicate that for CMSX-4 the  $K_1$ -concept may be used at both test temperatures.

### RESULTS

#### INCONEL 617 at 500°C

A comparison of the behaviour of creep crack either obtained by the evaluation as fatigue- or as creep-crack curves (Figure 2) shows: the values of  $\Delta K_I$  in air and in vacuum are similar, the specimen in vacuum, however, developed a much higher resistance for creep deformation with higher  $\Delta K_I$ - values. In the Paris regime of the crack curve the slope measured in both test atmospheres is similar. The behaviour of the crack growth curve in open air may be derived by a parallel transfer of the curve obtained in vacuum. The microstructure in the crack path consists of three regimes, whereby the fracture strain lines are observed to be stronger in open air than in vacuum.

After optimisation of the marker parameters [2,6], the marker lines are visible in the crack path surface. There are the same indications observed for this material at the test temperature. The crack initiation in air requires less stress intensity compared to vacuum; the Paris slopes are similar in both test environments, the end of deformability is higher in vacuum than in air (Figure 2).



Figure 2: Results of crack growth experiments with INCONEL 617 in air and vacuum at 500°C, measurements by potential drop (PD) or by the marker technique [6]

# Creep crack growth of wrought alloys

For the application of the wrought alloys INCONEL 617, INCONEL 706 and Waspaloy as large scale components, the creep crack growth behaviour becomes important. The results shown in Figure 3 demonstrate the influences of the temperature and of the environment on the creep crack growth resistance in the temperature range of 650 - 750°C. Figure 3a shows a comparison of the three alloys at 700 °C; for INCONEL 706, an  $\eta$ -free and an  $\eta$ -containing variant were investigated. The  $\eta$  phase precipitates as a cellular structure on the grain boundaries (see Figure 1) which results in an increase of the crack growth rate by a factor up to 10 compared with to the  $\eta$  free variant. Waspaloy shows the best creep crack growth resistance of the three alloys, with the highest K value for the crack initiation.

An example for the temperature dependence of the creep crack growth is given in Figure 3b. The exponent of the crack growth equation shows the highest value at 700 °C and the initial K value decreases with increasing temperature. The influence of the environment is demonstrated with the example of the  $\eta$ -free INCONEL 706 variant at temperatures of 650°C and 700°C in Figure 3c. The creep crack growth rate is higher in air than in vacuum, but with increasing temperature influence of test environment diminishes.



Figure 3: (a) Comparison of creep crack growth at 700°C in vacuum for INCONEL 617, INCONEL 706 (ηcontaining and η-free versions) and Waspaloy; (b) effect of temperature on creep crack growth rate of Waspaloy in vacuum; (c) effect of temperature and test environment on creep crack growth in ηfree INCONEL 706.

#### SEN CMSX-4 at 750 and 1000°C

The results of fatigue crack growth experiments with SEN CMSX-4 specimens are summarised in Figure 4. At 750 and 1000°C, the fatigue crack growth behaviour of specimens with different orientations resulted in the expected functional behaviour ("Paris-Erdogan") of da/dN versus  $\Delta K_I$  (the cyclic stress intensity factor [3]. At 750°C, the threshold values were higher and the slope of the Paris equation not so steep compared to the values at 1000°C. The influence of the crack path in an <001> oriented specimen seemed to be more marked at 750 than at 1000°C. Specimens with a <100> crack path orientation came to a sudden fracture by a spontaneous change to the {111} sliding planes. The <110> crack path orientation did not show this behaviour.

Figure 4 compares the fatigue and the creep fatigue behaviour at 1000°C. The edge crack specimen showed the same threshold values for both types of test, but the creep crack curves did not exhibit any changes in the crack growth rate. Therefore one may expect that creep-fatigue is more influenced by the deformation at the crack tip than by oxidation. If K as the stress intensity factor controlling the creep crack behaviour is used, the fatigue and the creep-fatigue results lie in the same range. At high K values and crack growth rates, the differences between fatigue and creep-fatigue became more significant. Because of these observations, one may conclude that creep dominates the crack growth process at low  $\Delta K$  or K values, and fatigue at high  $\Delta K$  or K values.



Figure 4 : Scheme of Crack growth behaviour of CMSX-4 in fatigue and creep fatigue crack growth tests [3].

Therefore, one may assume that the position of the crack front in relation to the {111} sliding planes is responsible for the observed brittle fracture. At 1000°C and for low  $\Delta K$  values and low crack growth rates, the crack growth behaviour may be understood as typical crack behaviour of small cracks. This behaviour could be explained by the crack closure because of plastic deformation at the crack tip, oxidation and depletion of the crack surface areas and the start of the  $\gamma'$  rafting process. These influences blunt the crack tip and the stress singularity decreases, so that the crack could be stopped or slowed down. At the low test temperatures, the influence of oxidation behaviour in the crack tip is not clearly demonstrated.

The oxidation behaviour at the crack tip became more important at the higher test temperatures. The comparison of the crack growth experiments in air and vacuum proved that the oxidation process influences significantly the crack initiation point or the initial stage of crack growth. Fractographic examinations using scanning electron microscopy (SEM) indicate for CMSX-4 (high volume fraction of  $\gamma'$ ) a slightly different behaviour compared to equiaxed Ni alloys with  $\gamma'$  volume fractions below 50%. In CMSX-4 at 750°C, high  $\Delta$ K-values and high rates of crack growth, the fracture surface tended to shift to a {111} gliding plane, whereas at 1000°C

this was not observed. The crack surface of CMSX-4 at 750°C air followed at low  $\Delta$ K-values the  $\gamma$  channels or the  $\gamma/\gamma'$  interface region. At high  $\Delta$ K-values, a change in the crack surface growth to be the {111} plane was observed and a cutting of  $\gamma'$  occurred.

# SUMMARY

The influences of test temperature and of the environment (air versus vacuum) have been investigated Ni-base alloys representing two different alloy types: solid solution hardened INCONEL 617 and the single crystal  $\gamma$ '-hardened CMSX-4. At test temperatures of 500 and 700°C there was no significant of test environment on the crack growth rate in the "Paris" region. However, a distinct influence of the environment on crack initiation was found.

The results of creep-fatigue crack growth experiments on the alloy CMSX-4, using single edge notched specimens at 750°C (maximum root-temperature) and for 1000°C (maximum airfoil temperature) showed that at 750°C and below the cracks, controlled by K concept, followed a zigzag line by changing the orientation along the  $\{111\}$  and  $\{100\}$  gliding planes. An influence of atmosphere was observed at the beginning and at the end of the crack growth. At high temperatures, such as 1000°C, crack propagation along the  $\{100\}$  planes was found.

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