Crack growth threshold under hold time conditions in DA Inconel 718 – A transition in the crack growth mechanism

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ABSTRACT. Aeroengine manufacturers have to demonstrate that critical components such as turbine disks, made of DA Inconel 718, meet the certification requirements in term of fatigue crack growth. In order to be more representative of the in service loading conditions, crack growth under hold time conditions is studied. Modelling crack growth under these conditions is challenging due to the combined effect of fatigue, creep and environment. Under these conditions, established models are often conservative but the degree of conservatism can be reduced by introducing the crack growth threshold in models. Here, the emphasis is laid on the characterization of crack growth rates in the low ΔK regime under hold time conditions and in particular, on the involved crack growth mechanism. Crack growth tests were carried out at high temperature (550 °C to 650 °C) under hold time conditions (up to 1200 s) in the low ΔK regime using a K-decreasing procedure. Scanning electron microscopy was used to identify the fracture mode involved in the low ΔK regime. EBSD analyses and BSE imaging were also carried out along the crack path for a more accurate identification of the fracture mode. A transition from intergranular to transgranular fracture was evidenced in the low ΔK regime and slip bands have also been observed at the tip of an arrested crack at low ΔK. Transgranular fracture and slip bands are usually observed under pure fatigue loading conditions. At low ΔK, hold time cycles are believed to act as equivalent pure fatigue cycles. This change in the crack growth mechanism under hold time conditions at low ΔK is discussed regarding results related to intergranular crack tip oxidation and its effect on the crack growth behaviour of Inconel 718 alloy. A concept based on an "effective oxygen partial pressure" at the crack tip is proposed to explain the transition from transgranular to intergranular fracture in the low ΔK regime.

KEYWORDS. DA Inconel 718; Fatigue crack growth; Hold time effect; Crack growth threshold; Intergranular to transgranular transition.

INTRODUCTION

Aeroengine manufacturers have to demonstrate that critical components such as turbine disks meet the certification requirements in term of fatigue crack propagation life, using damage tolerance approaches. Crack propagation laws are usually identified from sinusoidal wave shape fatigue tests. Trapezoidal wave shape signal tests, with a hold time at maximum load, are also studied as they are more representative of the in service loading conditions (i.e. the take off – cruise – landing cycle). This study aims at investigating the deleterious effect of hold time on the crack propagation behaviour of DA Inconel 718 [1-3], a polycrystalline nickel based superalloy widely used for aeroengines turbine disks manufacturing. Modelling the hold time effect is challenging as models have to take into account the coupled effects of fatigue, creep and environment (see e.g. [4-6]). Established models are often conservative. Introducing the crack growth threshold under hold time conditions in models is a way to reduce the degree of conservatism. This paper focuses on the characterization of the crack growth threshold under hold time conditions and crack growth mechanisms involved in the low ΔK regime.

Crack growth tests were carried out under hold time conditions using a K-decreasing procedure. This procedure, described in [7], is generally used to determine the fatigue crack growth threshold. Different hold times (300 s and 1200 s) have been investigated at temperatures ranging from 550 °C up to 650 °C. Studies regarding the crack growth threshold under hold time conditions are scarce, but the reader can refer to the work of Lynch [8] and the more recent work of Li [9]. In the present paper, the emphasis is laid on the characterization of the fracture mode involved in the low ΔK regime. Scanning electron microscopy (SEM) was used to characterize the transgranular or intergranular aspect of the fracture surfaces. Electron backscattered diffraction (EBSD) analyses were also carried out to observe the crack path at a microscopic scale in the low ΔK regime.

MATERIAL AND EXPERIMENTAL PROCEDURE

Material data

The material used in this study is the direct aged version (DA) of Inconel 718, a wrought polycrystalline nickel based superalloy widely used for aeroengines turbine disks manufacturing. The chemical composition of the material is presented in Tab. 1. The material was delivered in the form of a forged disk which was given the DA heat-treatment (720 °C for 8 h and 620 °C for 8 h) directly after forging. The DA Inconel 718 exhibits a γ matrix with a face-centered cubic structure and a small grain size of 5 to 15 µm. Two strengthening particles, the γ' (Ni_3 (Ti, Al)) phase and the metastable γ" (Ni_3Nb) phase, are precipitated inside the γ matrix. A large amount of stable δ (Ni_3Nb) phase particles are formed mainly along the grain boundaries. The δ phase is the stable version of the γ" precipitates, thus a γ" precipitates depleted zone is observed around δ particles giving rise to a local softening.

Element	Ni	Cr	Fe	Nb+Ta	Mo	Ti	Al	Co	Mn	Cu	C	B
Weight %	Balance	17.97	17.31	5.4	2.97	1	0.56	0.14	0.08	0.03	0.023	0.0041

Table 1: Chemical composition of DA Inconel 718.

Experimental procedures

Tests were carried out on KBr specimens with a rectangular cross-section of 8.3 x 3.5 mm². A semi-circular starter notch of depth 0.3 mm is introduced by EDM. Specimens were fatigue-precracked at 450 °C at a frequency of 10 Hz, using a K-decreasing method according to ASTM E-647 [7]. This was done to obtain a sharp semi-circular crack of 1.3 mm length with a reduced plastic zone ahead of the crack tip. Specimens were then heated up to the test temperature. Temperatures ranging from 550 °C to 650 °C have been investigated.

The first part of the test consisted in a fatigue crack growth test under load control conditions. The tests were performed using 10-X-10 trapezoidal load cycles, where 10 is the time in seconds used to load and to unload the sample, and X denotes the hold time (in seconds) at maximum load. Hold times of 300 s and 1200 s have been investigated. All the tests were performed at a constant load ratio of R=0.05. During tests, the crack growth was monitored using a direct current potential drop technique (DCPD) previously calibrated by finite element calculations (see for e.g. [10]). These testing conditions were applied until the crack propagates over 200 µm in order to collect data in the Paris regime.
The second part of the test focused on the low ΔK regime and consisted in a K-decreasing procedure performed by shedding the applied force step wisely while applying the investigated 10-X-10 cycle. ASTM E-647 recommends applying this method until a crack growth rate of 10^{-10} m/cycle or less is reached to determine the crack growth threshold. Doing so with 10-X-10 cycles would be time consuming and for this reason, tests were performed until crack growth rates between 10^{-8} m/cycle and 10^{-9} m/cycle were reached. Another fatigue crack growth test can be subsequently performed with hold time under load control conditions to acquire more data. On the other side, several tests were stopped directly after the K-decreasing procedure to obtain information at the crack tip in the low ΔK regime.
Scanning electron microscopy was used in order to determine the transgranular or intergranular nature of the fracture surfaces. Crack path observations were also performed. The samples (broken at the end of the test) were axially cut, perpendicularly to the fracture surface, with a diamond tip cutter. The crack path appears along the edge of the cut plane. In order to protect the crack path during polishing and to avoid blunting it, a nickel coating was applied on the fracture surface. This way, the blunting will affect the nickel coating edge and allow keeping the crack path undamaged. The crack path will then be observed using electron backscatter diffraction and back-scattered electron (BSE) imaging.

RESULTS

Fracture surfaces observations

Scanning electron microscopy was used to observe the fracture surfaces of samples tested under hold time conditions in the Paris regime first. For all the investigated temperatures (from 550 °C up to 650 °C) and hold time durations (300 s and 1200 s), the fracture surfaces exhibited an intergranular fracture (see Fig. 1-b). For the same temperature, under 2 Hz pure fatigue loading, transgranular fracture was observed (see Fig. 1-a). A large amount of δ phase particles were observed on both transgranular and intergranular fracture surfaces (indicated by white arrows on Fig. 1-a and 1-b).

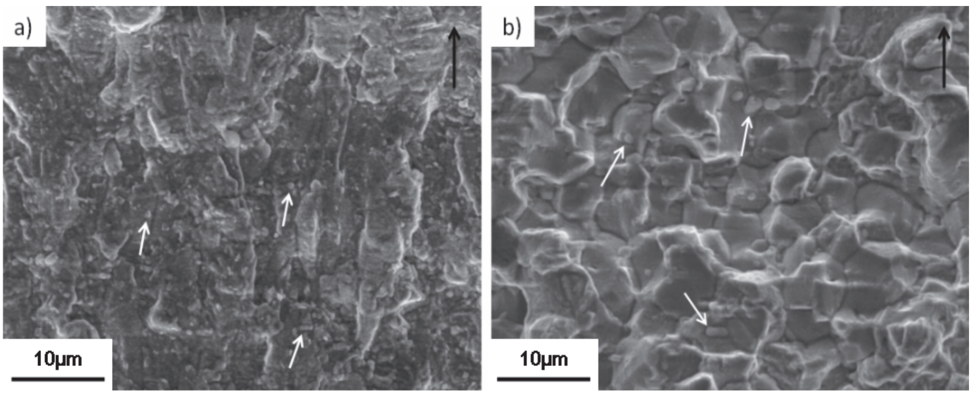

Figure 1: a) Transgranular fracture under sinusoidal 2Hz load cycle, b) Intergranular fracture under hold time conditions. The black arrows indicate the crack propagation direction.

The fracture surfaces in the low ΔK regime were also investigated. A progressive transition from a fully intergranular fracture (Fig. 2-b) to transgranular fracture (Fig. 2-c) was observed as K decreases. No fatigue striations were observed on the transgranular domain. Such a transition has also been observed by Li [9] in alloy 720Li, using a load increasing procedure. This procedure consists in applying the 10-X-10 cycle at a load level corresponding to a K slightly higher than the fatigue crack growth threshold ΔK_{th}. The maximum load level is then progressively increased until a significant crack growth is detected using the DCPD method. This method was also used on some samples. The load level was increased by 10 % every 24 h if no significant crack growth was detected as monitored with the DCPD method. Using this testing method, transgranular fracture was also observed on the fracture surface (see Fig. 3) before a fully intergranular fracture observed at higher ΔK. These observations confirm the results of Li [9] for DA Inconel 718.

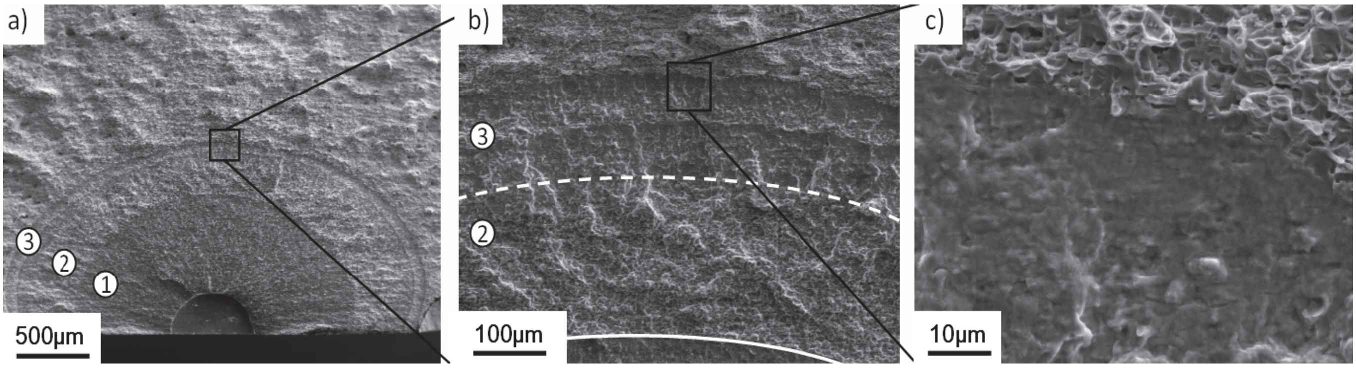

Figure 2: SEM micrographs showing the fracture surface of a specimen tested at 650°C with 10-300-10 cycles: a) 1 - fatigue pre-cracking, 2 - 10-300-10 propagation and 3 - 10-300-10 propagation in the low ΔK regime during the K-decreasing procedure. b) Transition from fully intergranular to transgranular fracture in the low ΔK regime. The solid line indicates the end of the fatigue pre-cracking step and the dashed line indicates the start of the K-decreasing procedure. c) Transgranular fracture at low ΔK.

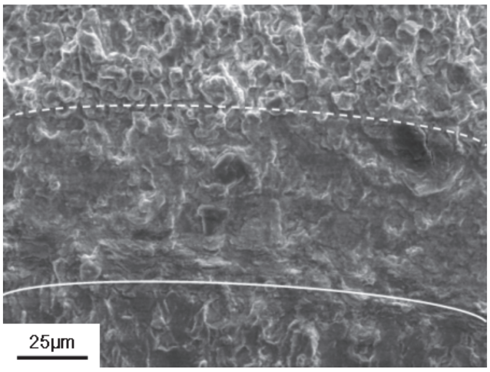

Figure 3: Transgranular fracture observed at low ΔK under 10-300-10 cycles at 600°C. The solid curve corresponds to the application of the 10-300-10 cycle, at low ΔK. The dashed curve indicates the fully intergranular fracture obtained at higher ΔK.

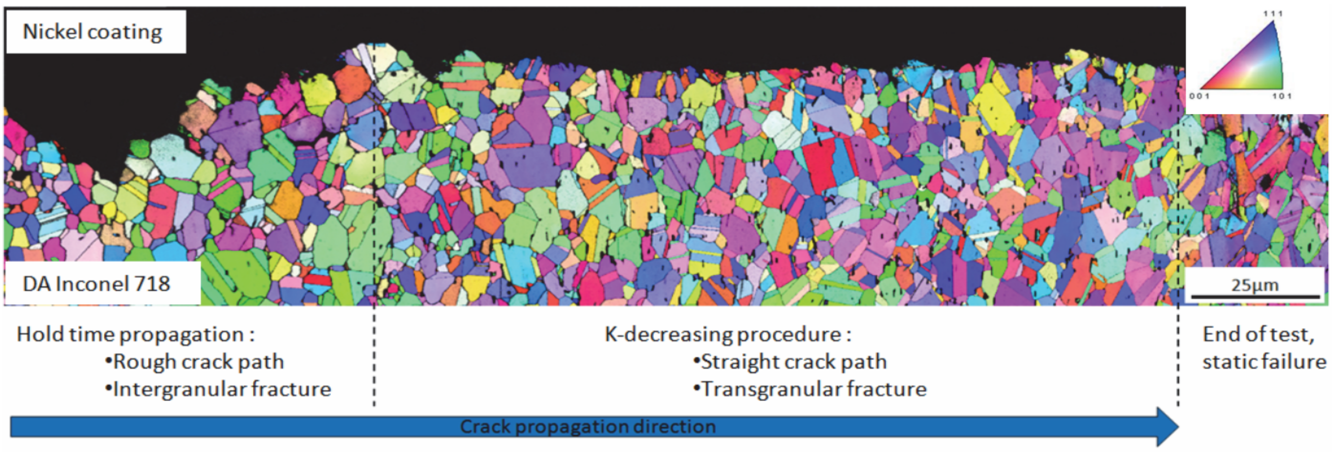

Figure 4: Inverse pole figure showing transgranular fracture along the crack path in the low ΔK regime. The nickel coating was removed from EBSD data by filtering data with the index quality of collected Kikuchi diagrams.

Crack path observations using EBSD

An in depth characterization of the crack path at a microscopic scale was performed using EBSD (Fig. 4). These analyses were performed to confirm the transgranular fracture in the low ΔK regime under hold time conditions, as it does not appear as a typical transgranular fracture mode, usually observed under 2 Hz pure fatigue loading (compare Fig. 1-a and

Fig. 2-c). EBSD analyses revealed a straight crack path and cut grains beneath the crack path, associated with transgranular fracture, in the low ΔK regime under hold time conditions. At higher ΔK, under the same loading conditions, the crack path appears rough with full grains beneath the crack path, indicating an intergranular fracture.
Characterizations of the fracture surfaces and the crack paths in the low ΔK regime revealed a transition from fully intergranular to transgranular as ΔK decreases, while cycling under hold time conditions. This has been observed at 650 °C for hold times of 300 s and 1200 s and at 600 °C for a hold time of 300 s. Below 600 °C, no transition was clearly observed. This transition is believed to be the sign of a progressive extinction of the embrittling effect of environment occurring during the hold time at maximum load, as K decreases. This transition is discussed in the next session, regarding the effect of environment and the mechanical state at the crack tip.

DISCUSSION

Introduction

It is well established, in Inconel 718 and other nickel-based superalloys, that the application of a hold time at maximum load has a deleterious effect on the crack growth behaviour (see for e.g. [1-3]). Under these conditions, the increased crack growth rates are accompanied with an intergranular fracture and the crack growth mechanism is time dependent. Without hold time at maximum load, and at higher frequencies, fracture is transgranular, lower crack growth rates are measured and the crack growth mechanism is cycle dependent.
The exact mechanism responsible for the embrittling effect of environment under hold time conditions is still not clearly understood but there exists two main theories. The stress assisted grain boundaries oxidation theory (SAGBO) [11] involves grain boundaries oxidation ahead of the crack tip and subsequent failure of these grain boundaries, of reduced fracture toughness, during the next loading. The dynamic embrittlement (DE) [12], instead, involves short distance oxygen diffusion in grain boundaries ahead of the crack tip. The embrittled grain boundaries will break during the next loading, exposing new surfaces to subsequent oxidation. In both cases, the embrittling effect of environment can be assumed as a process leading to reduced grain boundaries fracture toughness ahead of the crack tip, thus inducing intergranular fracture.
An explanation to the observed transgranular fracture under hold time conditions at low ΔK is proposed here, based on the coupled effect of oxidation and the mechanical state at the crack tip.

On the concept of grain boundaries fracture toughness

At a microscopic scale, the crack front can be considered as a population of grain boundaries interfaces with its respective orientation regarding the load axis. These interfaces will not break unless the applied loading excesses the grain boundaries fracture toughness. Whatever the damaging process occurring during hold time is, it can be assumed as a short distance embrittling process, consistently with the work of Molins [13], leading to reduced grain boundaries fracture toughness. This reduced grain boundaries fracture toughness may be due to the formation of oxides or the diffusion of embrittling species along the grain boundaries ahead of the crack tip. At a given temperature, and in the absence of any applied load, the grain boundaries fracture toughness can be assumed as an intrinsic property of the material. The grain boundaries fracture toughness may decrease during a sustained loading, due to the continuous embrittling effect of environment and the material ability to relax stresses. Then, it could be expected that temperature has a deleterious effect on the grain boundaries fracture toughness. This concept is illustrated in Fig. 5. Under hold time conditions, the grain boundaries fracture toughness will be reduced due to the embrittling effect of the environment, thus inducing intergranular fracture if the applied load excesses this fracture toughness.
This concept could explain the transgranular fracture observed in the low ΔK regime under hold time conditions. As K decreases, the applied mechanical load will become lower than the reduced grain boundaries fracture toughness, thus inducing transgranular fracture at low ΔK.

Discussion regarding the combined effect of the environment and the mechanical state at the crack tip

It was shown by Molins [13] that the embrittling effect of the environment is a dynamic coupling of the oxidation process and the mechanical state at the crack tip, especially plastic strain rates. By performing crack growth tests on Inconel 718 at different oxygen partial pressures, the author has demonstrated the existence of a transition pressure below which the environment has no effect on crack growth rates. Such a transition was also evidenced on N18 superalloy [14]. This transition pressure (between 10^{-2} and 1 Torr) is independent of the loading conditions. Further experiments were performed by superimposing a 60 s pressure cycle, above the transition pressure, over the mechanical cycle. This pressure cycle was applied at different instants of the hold time cycle (see Fig. 6). As the deleterious effect of the pressure cycle decreases along the hold time, it was then concluded that the effect of the pressure cycle is related to the strain rates at the crack tip as stress relaxation occurs during the hold time.

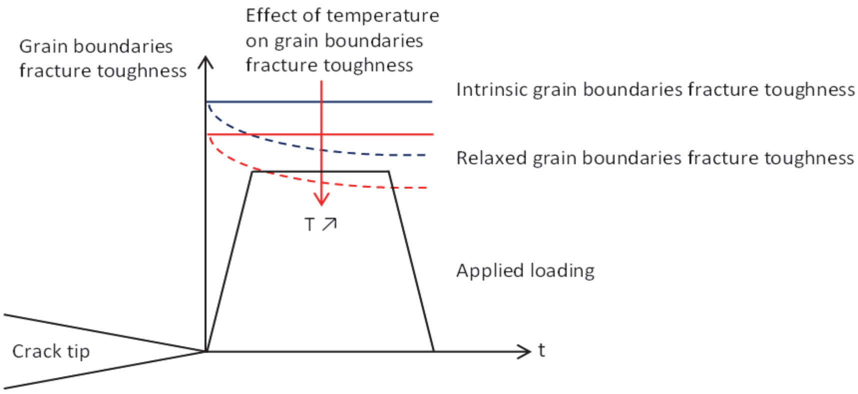

Figure 5: Schematic illustration of the grain boundaries fracture toughness concept, applied at the crack tip.

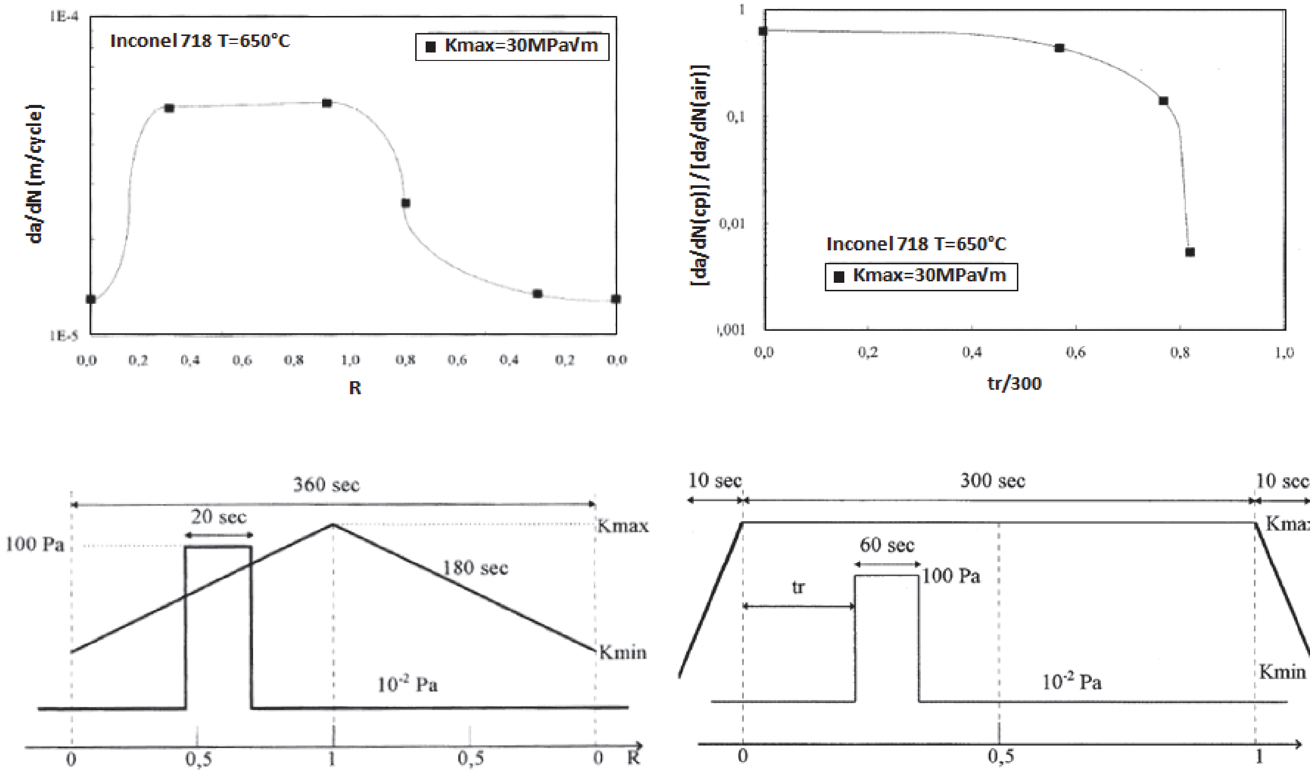

Figure 6: Results on Inconel 718 at 650°C showing the part of the loading cycle where environment has an effect on crack propagation [13].

During the K decreasing procedure, the strain rate at the crack tip decreases. Consistently with the work of Molins [13], once the strain rates are lower than a critical value, the environment has no effect on the crack growth rates. As K decreases, it is assumed the embrittling effect of environment occurring during the hold time progressively vanishes. The hold time cycle can progressively be considered as an equivalent pure fatigue cycle of frequency leading to a cycle dependent crack growth mechanism, consistently with the observed transgranular fracture at low ΔK.
BSE imaging was performed at the tip of an arrested crack at low ΔK (ΔK≈13 MPa√m), tested under hold time conditions (see Fig. 7). The crack path appears partially transgranular and slip bands are observed at the crack tip. This is usually observed under pure fatigue loading conditions and tends to indicate a crack propagation mechanism governed, at low ΔK under hold time conditions, by cyclic plastic strain at the crack tip.

To confirm the assumption of an equivalent pure fatigue cycle at low ΔK, tests will be carried out with a continuous monitoring of the crack length during the hold time, using the DCPD method. This way, the crack growth occurring during the hold time could be isolated from the crack growth occurring during the cyclic part of the cycle. The confirmation would be achieved if no more crack growth during the hold time is detected at low ΔK.

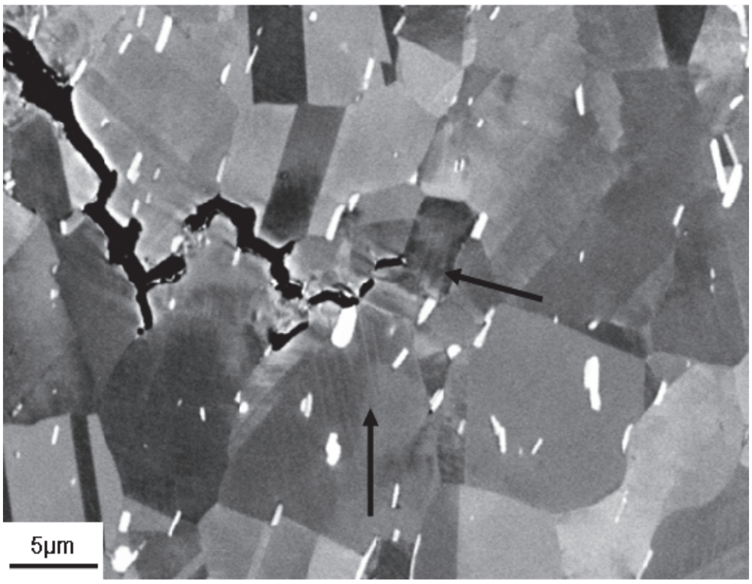

Figure 7: BSE imaging of the crack tip region (ΔK≈13 MPa√m). Slip bands (indicated by black arrows) can be seen at the crack tip and the crack path appears partially transgranular. Test conditions: K-decreasing procedure under 10-300-10 loading at 600 °C.

The framework of the analysis appears consistent with the observed transition from intergranular to transgranular fracture at low ΔK under hold time conditions. Characterizations carried out at the tip of an arrested crack at low ΔK tend to confirm the assumption of an equivalent pure fatigue cycle at low ΔK. However, this analysis does not take into account the embrittling mechanism of the environment occurring during the hold time.

On the concept of an "effective oxygen partial pressure" at the crack tip

It was demonstrated by Andrieu [15] that the transition pressure, described above, is accompanied by a transition in the fracture mode. Below the transition pressure, transgranular fracture is observed, while it is mainly intergranular at higher pressure. It is also demonstrated that oxidation at the crack tip is governed by the operating oxygen partial pressure. Above the transition pressure, Ni and Fe rich oxides will form first. This porous oxide layer leads to a reduced partial pressure at the metal-oxide interface, then leading to the build-up of a protective Cr_2O_3 layer. At lower pressure, only the protective Cr_2O_3 layer will form.

The intergranular fracture, obtained at high partial pressure, could be explained by the deleterious effect Ni and Fe rich oxides may have on the grain boundaries fracture toughness ahead of the crack. The reduced grain boundaries fracture toughness will become lower than the applied mechanical load at the crack tip, inducing intergranular fracture.

Regarding this result, one could explain the observed transition from intergranular to transgranular fracture in the low ΔK regime by the concept of an "effective oxygen partial pressure" at the crack tip. All tests presented in the first section were carried out in laboratory air. As K decreases, the effective stress intensity factor range ΔK_{eff} decreases and consequently the crack tip opening displacement (CTOD) too. The lower the CTOD, the less oxygen would be available at the crack tip due to a trapping effect by the crack lip, resulting in a lower oxygen partial pressure at the crack tip. The effective oxygen partial pressure at the crack tip may decrease to the extent it will become lower than the transition pressure. The Ni and Fe rich oxides will not form at the crack, then preventing the grain boundaries fracture toughness decrease and subsequent intergranular fracture. Assuming this concept, one could expect to observe only Cr_2O_3 on the transgranular fracture surface observed in the low ΔK regime.

Moreover, as shown in Fig. 7, slip bands were observed at the tip of an arrested crack in the low ΔK regime (ΔK≈13 MPa√m), under hold time conditions. This is believed to be a sign of a transition from a time dependent to a cycle dependent crack growth mechanism. These slip bands can act as rapid diffusion paths for Cr [15], thus allowing a sustained Cr supply at the crack tip. This would lead to the preferential build-up of a protective Cr_2O_3 layer. To confirm this assumption and the proposed concept of an effective oxygen partial pressure at the crack tip, characterizations of oxides formed on the transgranular fracture surfaces, in the low ΔK regime will be carried out.

CONCLUSION

This work aimed at studying crack propagation in the low ΔK regime, under hold time conditions in DA Inconel 718 alloy. Crack growth test were carried at high temperature using a K-decreasing procedure. The results presented in this paper focuses on the fracture mode and the crack growth mechanisms. Based on SEM observations of the fracture surfaces and EBSD analyses along the crack path, a transition in the crack growth mechanism is evidenced in the low ΔK regime. Results and proposed analyses can be summarized as follow:

1. In the low ΔK regime, transgranular fracture was observed on the fracture surfaces and along the crack path on samples tested under hold time conditions at maximum load. Under the same loading conditions, and at higher ΔK, the fracture is intergranular. Transgranular fracture is usually observed on fracture surfaces of sample tested under pure fatigue loading conditions, at higher frequency.
2. Slip bands have been observed at the tip of an arrested crack tested under hold time conditions at low ΔK (ΔK≈13 MPa√m). This is usually observed under pure fatigue loading conditions and indicates a crack propagation mechanism governed by cyclic plastic strain at the crack tip. This, associated with the transgranular fracture, could be explained assuming the hold time cycle acts, at low ΔK, as an equivalent pure fatigue cycle.
3. To explain the transition from intergranular to transgranular fracture as ΔK decreases, the concept of an effective oxygen partial pressure at the crack tip is proposed. This could be sustained by an oxygen trapping phenomenon at the crack tip, related with reduced CTOD in the low ΔK regime. Lower oxygen partial pressure at the crack may favour the build-up of a protective Cr_2O_3 oxide layer. This would prevent the formation of Ni and Fe rich intergranular oxides at the crack tip. These oxides have a deleterious effect on the grain boundaries fracture toughness, then leading to intergranular fracture. Preventing the formation of these oxides would prevent intergranular fracture and thus explaining the transgranular fracture observed in the low ΔK regime. Furthermore, the presence of slip bands at the crack tip allows the rapid diffusion of Cr toward the crack tip, thus favouring the build-up of a protective Cr_2O_3 oxide layer.

Further experiments will be carried out to confirm the proposed mechanisms to explain the observed transition to a transgranular fracture at low ΔK. Crack growth tests using the K-decreasing procedure will be carried out with a continuous DCPD monitoring of the crack growth. This way, crack growth occurring during the hold time can be separated from the crack growth occurring during the cyclic part of the cycle. The crack growth occurring during the hold time is expected to progressively vanish at low ΔK. This way, the assumption of an equivalent pure fatigue cycle could be confirmed. Regarding the proposed concept of an effective oxygen partial pressure at the crack tip, in depth characterization of oxides formed on the fracture surfaces will be carried out. The proposed concept will be confirmed if different oxides layers are observed on the transgranular domain, corresponding to the low ΔK regime, and on the intergranular domain.

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