

## Fracture mechanisms during intergranular hold time fatigue crack growth in Inconel 718 superalloy.

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**Abstract** Ni-base superalloy IN718 is known to display time-dependent intergranular crack growth under dwell time mechanical loading at high temperature under atmospheric conditions. Oxygen has been pointed out as a cause of the intergranular damage causing embrittled crack growth during both cyclic and hold time loading. Investigation of the mechanisms responsible for the embrittlement should not only focus on the effect of environment but also on the combined action of fatigue, creep, temperature and time. In this work material from experiments with fatigue crack growth in combination with hold times of different length at different temperatures has been investigated. Fractographic studies and metallographic cross sections of fatigued specimens has been subjected to careful analysis using ECCI- imaging in order to shed light on the fracture mechanisms. The results show that the damage is caused by the influence of a combination of environment and severe local damage manifested as a transformation of the microstructure into sub cells, micro twins and recrystallised areas close to the crack tip. The damage mechanism is thus influenced by a combination of oxidation and severe local plastic deformation.

**Keywords** Intergranular damage, Dwell time, ECCI, Fatigue, Fractography

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### 1. Introduction

Ni-base superalloys are widely used in high temperature and for applications like rotating discs in gas turbines where IN718 is a popular alloy due to relatively low price/performance ratio, good corrosion resistance and mechanical properties with excellent weldability. The high temperature performance is limited to 650°C and an increase in temperature needed to increase efficiency has created demands of increasing temperature in steps of 50°C. Since IN718 has a strengthening phase of DO<sub>22</sub> based on Ni and Nb called  $\gamma''$  the max temperature is 650°C while a similar precipitate based on Ni and Al ( $\gamma'$ ) dissolves at a higher temperature. A newly developed  $\gamma'$ -former called Allvac718plus [1] is a replacement candidate for IN718.

The Ni-base superalloys are during service subjected to a combination of static loading and fatigue loading controlled by stress as well as strain. In addition to that high temperature and environment will act to reduce service life. The growth of fatigue cracks will be affected by temperature and environment so that the mode of crack growth is shifted from transgranular to intergranular. This embrittlement effect that most superalloys have in common [2] has been analysed in a great number of publications [3-8] and reviews [9-11] [12]. The fact that not only fatigue fracture crack growth is influenced by, frequency, temperature and environment but also growth of a static crack in conjunction with fatigue has been subject to analysis [13, 14]. The conclusions have so far been that the significance of the effect is minor under present service conditions but with increasing service temperature and load the effect might be a serious matter for the future.

The aim of this work is to study the effect of growth of fatigue cracks at high temperature in IN718 and Allvac718Plus in order to better understand the mechanisms behind the growth of fatigue

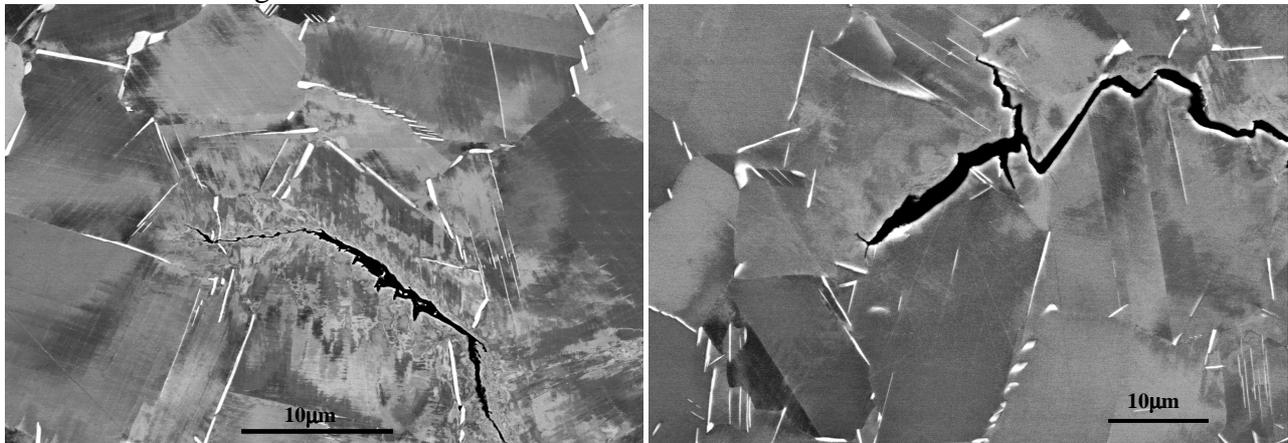
cracks especially dwell time cracking at hold times with varying length.

## 2. Experiments

Two alloys IN718 and Allvac718Plus have been subjected to fatigue testing with dwell time. The testing was performed in servohydraulic testing machines at constant stress amplitude using SEN specimens [8] with DCPD crack monitoring at high temperature using a radial furnace or induction heating [8, 15, 16]. Specimens with a crack were studied either as cross sections or as fracture surfaces using an Hitachi S70 analytical SEM with an annular Back scatter detector making it possible to produce images by Electron Channeling Contrast Image (ECCI). This method has been used with great success to study damage mechanisms in super alloy single crystals [17, 18]. The studied specimens have been chosen from crack growth experiments performed with Kb-type of specimens with cross sections of 4.3 X 10.2 mm with a 0.2 mm sparac cut starter notch. A baseline series of specimens were run at 0.5 Hz, R=0.005 and the hold time testing (90s, 2160s and 21600s) was performed at 450, 550, 650 and 700°C in lab air environment except for Allvac718Plus that was also tested in pure oxygen atmosphere and the results presented as crack growth curves in ref. [5, 16, 19-21].

## 3. Results

In order to be able to distinguish between crack growth at different temperatures with and without dwell the results from crack growth without dwell will be presented first. In figure 1 the crack zone for baseline cracking at 450°C and 650°C is shown.



a

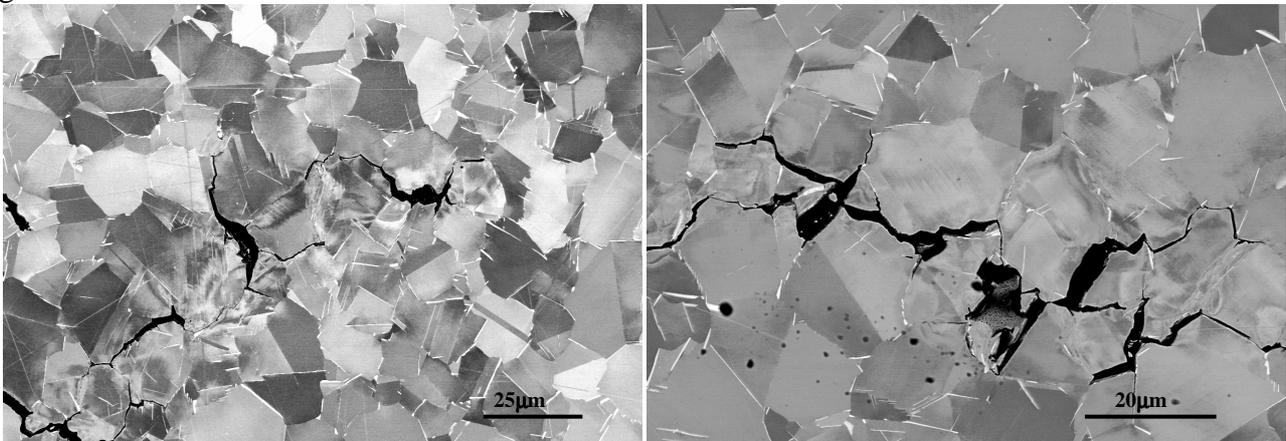
b

Figure 1. SEM-ECCI micrograph showing cross section of the crack tip region of IN718 subjected to base line fatigue at a) 450°C showing high slip activity and 650°C with lower slip activity.

The lower temperature shows clear channeling contrast from plastic deformation caused by homogeneous slip on primary slip systems and in a narrow zone close to the crack localized more concentrated multiple slip. The crack path is transcrystalline with slight crack branching. The crack growth mechanism seems to be based on a weakening of interface at the crack tip by plastic deformation. For the higher temperature the crack path is more intergranular with more crack branching and the slip intensity is less pronounced.

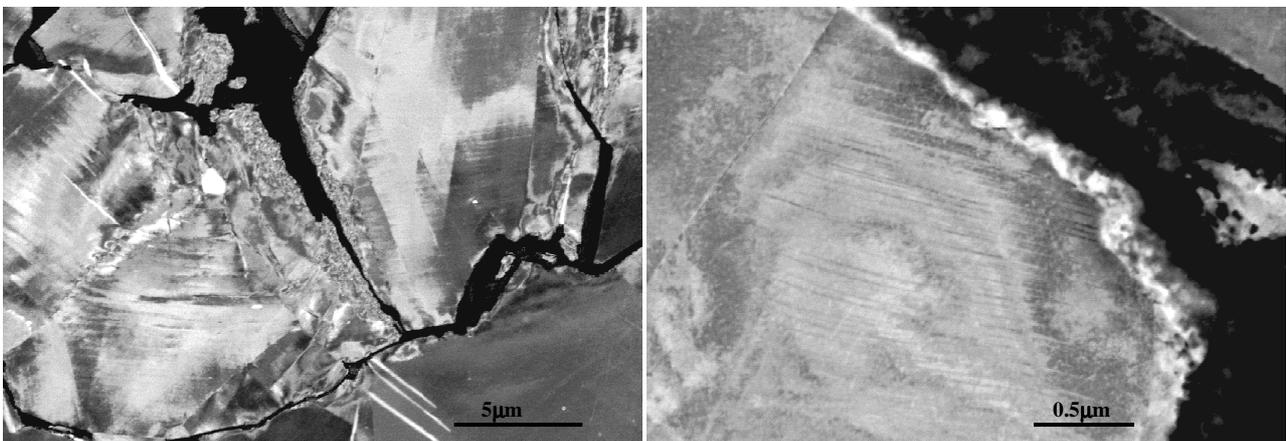
The specimens subjected to dwell show more intergranular fracture and branching Figure 2 and the plastic deformation is more pronounced at 550°C but the fracture mode is varying along the crack

path and the crack growth seems to be assisted by both slip, plasticity induced dynamic recrystallisation sometimes with twins or formation of voids and cracking oxide at the crack tip. Although the crack growth is considered intercrystalline it can be divided into three different growth modes.



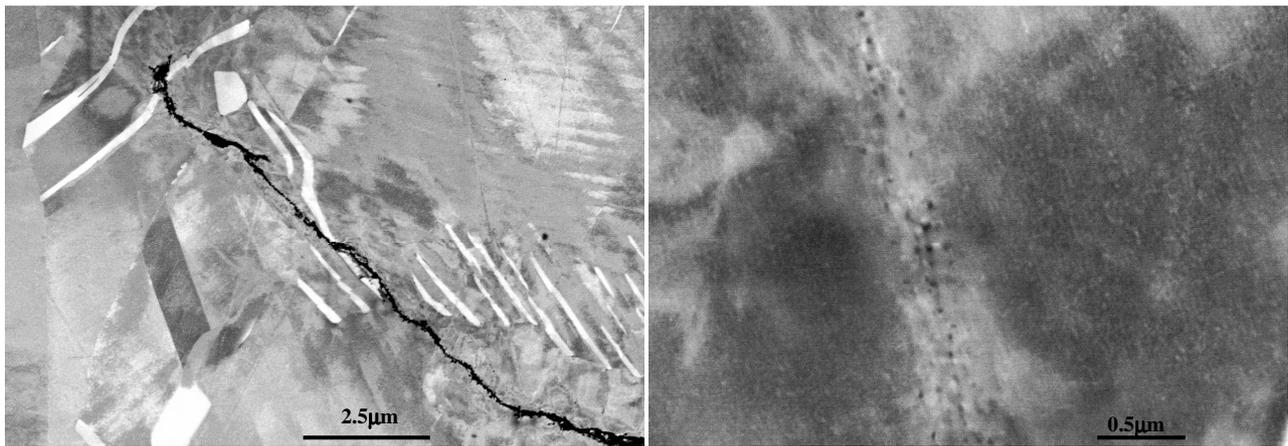
a b  
Figure 2. SEM-ECCI micrographs showing cross sections of specimens with hold time at a) 550°C with intergranular branched cracks and clear signs of slip and b) 650°C with intergranular cracking and less pronounced plasticity.

The first one is the growth along grain boundaries where the mating grains are subjected to planar slip on one or two slip systems Figure 1a. The second growth mode is along grains with heavier homogeneous slip and a narrow zone close to the fracture surface with heavy non planar dislocation structure and sometimes twins and recrystallisation structure nicely shown in ECCI mode in the SEM Figure 3.



a) b)  
Figure 3. SEM-ECCI micrographs showing a) the plastically damaged zone with sub grains and beginning recrystallisation close to the crack surface and b) deformation twins.

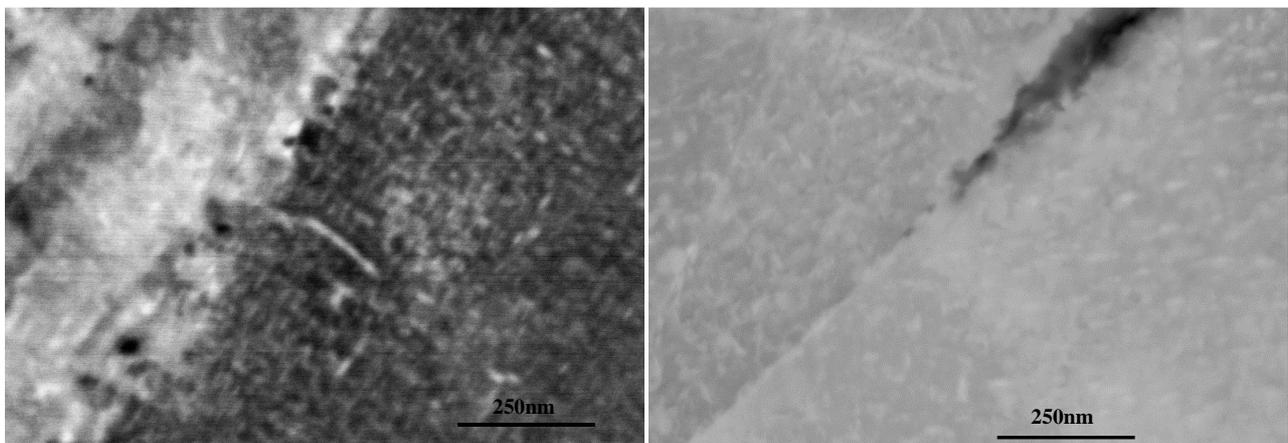
The third growth mode is cracking along  $\delta$ -phase often oxidized into a sponge like structure in the interface between a  $\delta$ -plate and the crack. It is thus difficult to say anything about the time-sequence of these events but the nanometric oxidized pores present at crack tips with a smallest size in the order of the hardening precipitates could trigger the onset of intercrystalline failure and the stress state and dislocation structure contributing factors Figure 4, 5, 6. The separate dwell time cracking



a

b

Figure 4. SEM-ECCI micrograph showing a) the crack tip region and b) the damage zone in front of the crack tip at higher magnification with stringers of pores.

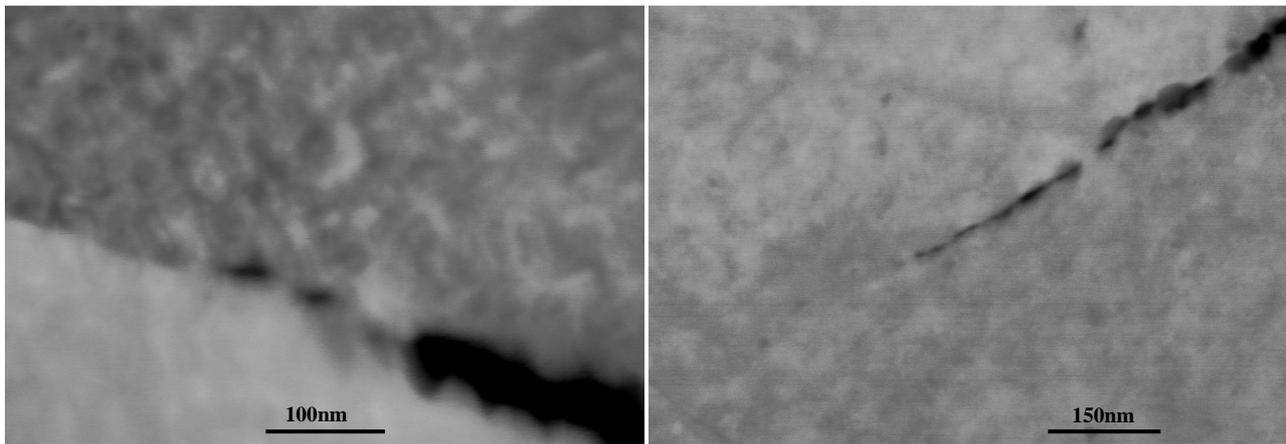


a

b

Figure 5. SEM-ECCI picture of the damaged zone in IN718 at 550°C temperature close to the crack showing a) the hardening precipitates along with nano sized pores in an area with high plastic deformation and b) pores in a grain boundary with no plastic deformation.

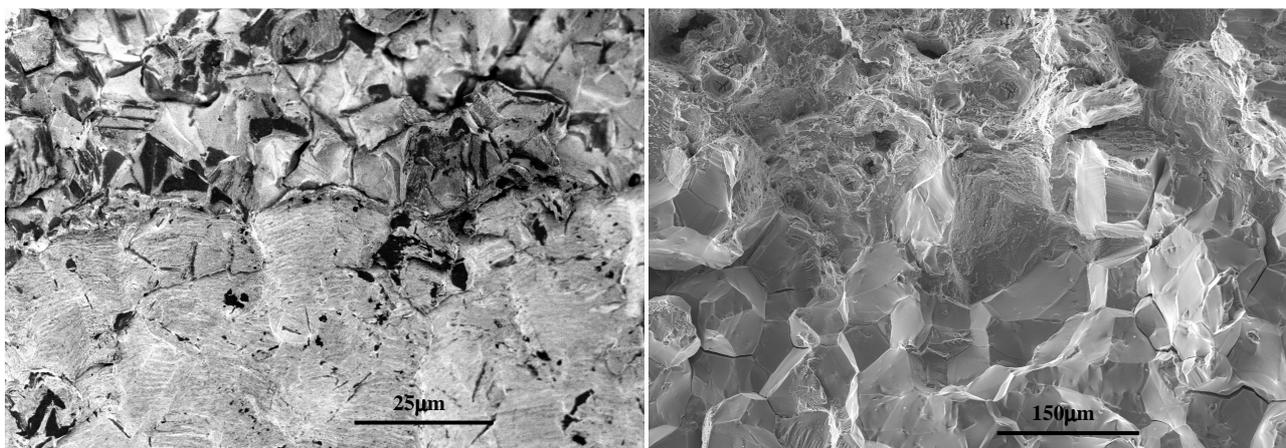
events caused by unloading between each hold time are difficult to distinguish along the crack front. The crack front seems to have the character of a spherical discontinuous process zone (fig.2) with branched intergranular cracks rather than a single crack moving through the microstructure. Crack growth could then consist of environmental oxygen induced intercrystalline growth of cracks along sensitized paths in different directions and an interlinking between embrittled areas showing ligaments with more severe slip. The ligaments are severely plastically deformed to a level that is very close to dynamic recrystallization with characteristic pores shown as black dots in Fig. 4b, Fig. 5 indicating a future crack path. The growth of the main crack is also influenced by interaction with  $\delta$ -plates acting either as a crack path or as a crack stopper (fig 4a) depending on orientation. The fact that the  $\delta$ -phase is ductile makes it less probable as a crack path but the interface between  $\delta$  and matrix is often acting as a crack [10] path through severely oxidized pores in the interface as observed in this work. Non propagating branched cracks are stopped due to blunting and/or geometrical reasons like going into mode II. Residual stress built up during growth manifested by a closure of the crack along the front also plays a role for the path taken by the crack.



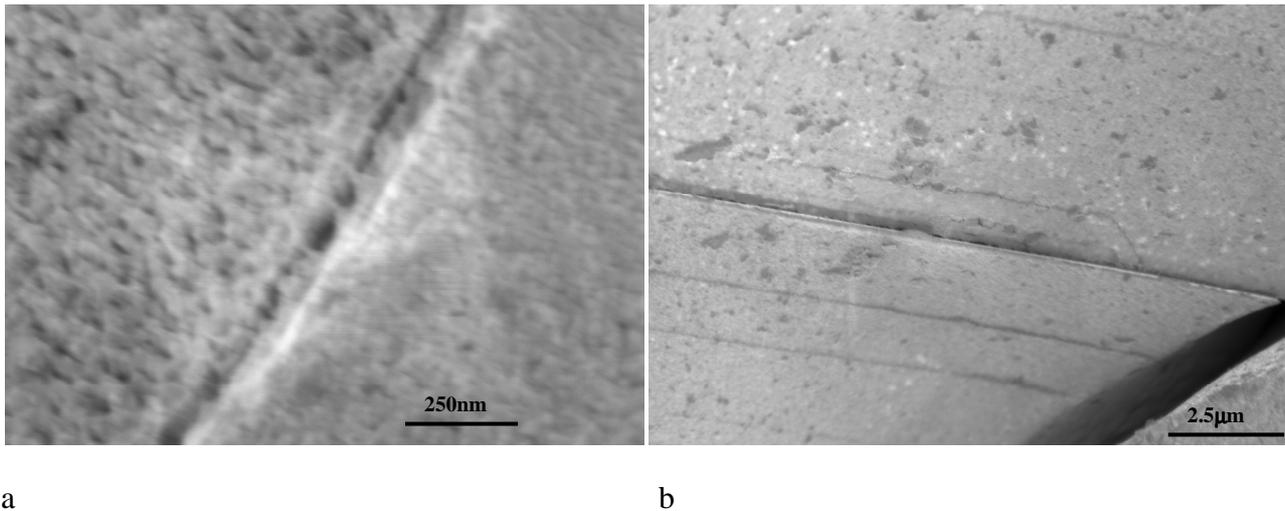
a b  
Figure 6. SEM-ECCI micrographs of a) IN718 650°C during hold and b) ALVAC718plus 700°C during hold. Both alloys show voids in front of the crack tip. The smallest voids are in the same range of size as the hardening precipitates that are clearly visible in a).

The macroscopic crack path often shows a mismatch orthogonally indicating shear or grainboundary sliding along the crack growth direction. SEM ECCI pictures taken at a very high magnification of non propagating secondary cracks is shown in Fig. 6 where nanometric voids in the same size range as the clearly visible hardening precipitates Fig 6a are seen along the grain boundary. This type of voids is often seen along the crack front for longer cracks being coarser and more oxidized.

The dwell time cracking of Alvac718Plus in pure oxygen atmosphere shows (Fig. 7b) more clearly the intercrystalline character of the crack growth due to the absence of  $\delta$ -plates (black in Fig. 7a) in this condition. The observable environmental action manifested by growth of oxidized pores in the grain boundaries seems to be the same as for IN718. The completely flat grain boundary surface has still slip bands and microscopic pores. The fatigue cycling in oxygen atmosphere at 700°C before the start of the dwell time cycle show clear striation like arrest markings indicating a continuous crack growth mode. Studying the fracture surfaces in higher magnification show signs of nano sized voids at the tip of branched cracks. The intergranular facets giving a smooth appearance at lower mag. (Fig 7b) show signs of voids at higher magnification (Figure 8, 9).



a b  
Figure 7. SEM fractographs showing a) boundary between pre cracking at room temperature (lower) and dwell time growth at 550°C (upper), black contrast is  $\delta$ -phase b) boundary between dwell cracking (lower) and final fracture (upper)in Allvac718plus at 700°C in pure oxygen atmosphere.



a b  
Figure 8. SEM fractographs of grain facets in Allvac718plus (700°C dwell) a) oxidized surface with voids b) surface at lower magnification with one open and one closed secondary crack with voids.

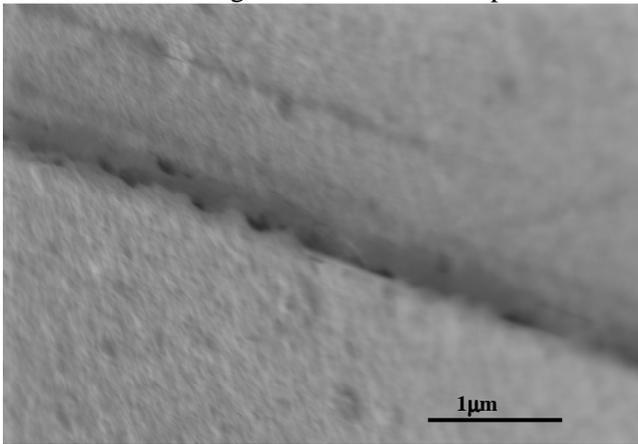


Figure 9. SEM fractograph showing the voids in the grain boundary shown in fig. 8b at higher magnification.

#### 4. Discussion

The results from studies of the crack growth process of IN718 and Allvac718Plus under fatigue and dwell time loading conditions at high temperature will be discussed. The focus will be put on the effects of events taking place during the hold time periods.

The crack growth under the influence of temperature and cyclic loading is transgranular at 450°C with clear evidence of planar slip in the grains surrounding the crack going over to intense slip close to the fracture surface with phenomena like nano twins and local recrystallisation appearing. The crack path is sometimes branched and not as transgranular as during room temperature cycling indicating that the grain boundaries play a role even during base line cracking. With increasing temperature the crack path becomes more branched and the plastic deformation is less pronounced. This indicates that the environmental effect is gradually increasing with temperature.

The hold time experiments consist of applied constant load cycles between 90 to 21660 s where each cycle is separated by unloading to zero load level. The observed macroscopic change in growth rate caused by those events can unfortunately not be traced on a micro level. The reason for that is probably that the crack advance is a non continuous process. Gas phase embrittlement, GPE by oxygen is by far the most proposed damage mechanism in this case [12]. Still very little is

known about the damage on a micro scale. The conditions at the crack tip are probably controlled by the environment and the diffusion of oxygen towards and into the grain boundaries. This process is in turn controlled by the conditions at the grain boundary where slip bands, local stress, voids, oxide formation and local chemistry will affect the diffusion of embrittling elements like oxygen. Depending on the local condition at the grain boundaries micro cracks will form and propagate till they are interfering and sometimes arrested by microstructure like  $\delta$  phase or blunted at different locations in different directions creating a damaged volume where the final crack path will be formed when the ligaments between the micro cracks will break after plastic deformation or embrittlement. This crack model can explain the apparent non continuous crack growth and all the other phenomena like oxidized voids and plastic deformation together with embrittlement. Crack closure caused by local plasticity and observed mixed mode character along the crack path may also play a role both due to the unloading cycle between the cycle but also due to plasticity and oxide formation[10]. The environmental crack propagation during dwell at 700°C in Allvac718plus is observed as close to purely intergranular with limited fractographic information of microscopic direction of growth while the fatigue crack growth during the same conditions show clear crack arrest markings during intergranular growth. The fact that there are virtually no crack arrest markings present during dwell does unfortunately not answer the question if the crack growth is continuous or not. Analysis of the crack tip with [15] advanced methods (SIMS) has shown that the penetration of oxygen is very limited in the crack tip region in front of the crack tip so the triggering of the crack growth event is probably caused by oxidation and growth of nano sized voids in the crack tip region. The voids can either be pure creep nano voids or  $\gamma''$  or  $\gamma'$  particles from the matrix identified in this study. The origin of the voids found in this study could be from  $\gamma''$  and/or  $\gamma'$  growing under the influence of local stress and dislocation activity [10] and subsequently close to the crack tip being opened up and oxidized to form the observed pores. The oxidation of  $\gamma''$  is probable in this environment since the  $\delta$ -plates exposed to oxygen, with the similar composition as  $\gamma''$  or even sometimes could be transformed to  $\delta$  by dislocation particle [11] shearing, have been observed to be severely oxidized. Since the analysis with SIMS has shown that diffusion of oxygen in front of the crack tip is nonexistent a growth mechanism based on oxygen diffusion must be extremely local in character. This supports a mechanism where nanometric voids are created by a combined action of creep, transformation/oxidation of  $\gamma''$  or  $\gamma'$  into pores where the pore walls are thin enough to allow transportation of oxygen before they break and gradually open up the crack. In a review by Woodford [12] the cavities are supposed to be created by oxidation of carbon into gas phase where the voids are created from gas bubbles. The bubbles are not only acting as creep cavities but also to reduce the grain boundary sliding that normally takes place to relieve stress concentrations in the grain boundary at higher temperatures. This is not contradicted by the observations in this work and could also explain the lack of expected oxide in front of the crack tip and also the fact that no oxygen at all was found by SIMS in ref. [16]. This also supports the observations in this work that there is a large amount of plastic deformation involved together with intergranular fracture in IN718 and no signs of embrittlement since the oxygen is not primarily causing embrittlement according to this model. Unfortunately evidence of a possible carbon source for the creation of gas bubbles to support the model proposed in Woodford has not been observed in this study but on the other hand not specifically been a subject of study.

Generally the growth of a crack during hold time constant loading is for IN718 a result of a number of growth mechanisms where microstructure together with plastic deformation and environment preferably oxygen are acting. For Allvac718Plus the dwell time effect at high temperature is more clear with pure intergranular growth mode but the growth mechanism during intergranular growth is very similar to IN718 with signs of oxidized pores in the [11] crack tip region.

## 5. Conclusions

A study of the growth of hold time cracks in IN718 and Allvac718Plus at high temperatures has led to the following conclusions.

- Growth of cracks in IN718 during dwell time show a mix between different growth modes with a marked shift in crack growth mode towards intergranularity with branching compared to fatigue crack growth under the same conditions.
- The growth mode during dwell time cracking is shifting towards more intergranular with increased branched cracking with increasing temperature for IN718 but local plasticity is still present.
- The dwell time cracking mode for Allvac718Plus is pure intergranular at high temperature with less pronounced local plasticity.
- Intergranular nanosized voids observed close to the crack tip region could be the responsible for intergranular cracking in both alloys.
- The origin of the nanosized voids is either pure mechanical or a result of oxidation of grain boundary precipitates

### Acknowledgements

The authors wish to great fully acknowledge Turbo Power consortium, Siemens Industrial Turbines, Volvo Aero Corporation. Agora Materiae and Strategic Faculty Grant AFM at Linköping University are also acknowledged.

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