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Effects of microstructure on high temperature dwell fatigue crack growth in a coarse grain PM nickel based superalloy

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Abstract — The influence of microstructure on the dwell fatigue crack growth behaviour of an advanced nickel-based superalloy was investigated at a temperature of 700 °C. Microstructural variations were induced by heat treatment variables: different cooling rates of quenching from super-solvus solution heat treatment, 0.7 and 1.8 °C s−1, and an addition of a high temperature stabilisation heat treatment (2 h at 837 °C) between the solution treatment and the final ageing treatment. With a one hour dwell introduced at the peak load of the fatigue cycle, such different microstructural conditions can lead to a difference of up to two orders of magnitude in crack growth rates in air, when compared to those obtained under baseline fatigue loading. By performing such dwell fatigue and baseline fatigue tests in vacuum, it is confirmed that such increases in crack growth rates under dwell fatigue loading in air are mainly environmentally related. Transmission electron microscopy (TEM) was utilised to analyse both crack tip oxidation and associated deformation mechanisms in the matrix. A novel mechanism taking into account competing interactions of crack tip oxidation (leading to increases in crack growth rates) and stress relaxation (leading to decreases in crack growth rates) is outlined.

Keywords: Environmental effects; RR1000; Dwell fatigue crack growth; Micromechanisms of crack growth; Grain boundary oxidation

1. Introduction

The relentless drive for lighter and more efficient gas turbine aero-engines has resulted in the development of a new generation of superalloys for turbine rotor disc applications. These highly alloyed materials, usually processed through a powder metallurgy (PM) route, have significantly higher proof stress and creep resistance at high temperatures than earlier superalloys, and these properties derive largely from having a high volume fraction of the intermetallic phase, γ (nominally Ni3Al). RR1000 is one such alloy, and is designed to operate at temperatures above the limits of other disc alloys such as Waspaloy a and UDIMET 720Li b [1]. RR1000 is commonly used in a fine grained microstructural condition, which possesses a good balance of high strength, high creep resistance and resistance against fatigue crack initiation and/or propagation. A coarse grained version of the alloy has potential to offer improved creep resistance and dwell fatigue crack growth resistance which are critical for the rim location of high pressure turbine discs where the temperature is at its highest [2].

With regard to dwell fatigue crack growth behaviour of nickel based superalloys, the influence of an oxidising environment has long been recognised. An increase in crack growth rates is generally observed at temperatures in excess of 500 °C, and such increases can be several orders of magnitude higher than those observed both in an inert environment and from “pure” fatigue (shorter dwell time, higher frequency) loading [3–10]. Acceleration of the rate of crack growth is often associated with a change in crack growth micromechanism and a transition in crack path from transgranular to intergranular. Such transitions are frequently interpreted in terms of superposition of time dependent crack growth either by creep and/or environmental damage which promotes crack growth along grain boundaries [11]. Effects of creep deformation and creep damage on crack growth should be considered carefully. Creep damage by cavity nucleation and linkage at grain boundaries can lead to accelerated crack growth [12]; while creep strain accumulation (deformation) can give rise to stress relaxation ahead of the crack tip. Such stress relaxation can potentially lower ‘effective’ mechanical driving forces and may therefore play a beneficial role in reducing rates of dwell (fatigue) crack growth [13]. Many factors have been

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observed to affect the crack growth behaviour including: temperature, microstructure, grain boundary chemistry and morphology, alloy chemistry, and loading frequency and waveform. Large variations in crack growth behaviour have been reported in the literature and a comprehensive understanding of the environmental effect is not yet available.

Two representative mechanisms are proposed in the literature to account for the effects of environment. Key arguments address whether it is an oxygen species or oxide film that is responsible for the weakening of grain boundaries and, hence, whether there is a long range transport of oxygen that can result in the formation of a sizeable oxygen-affected region. OAR \cite{14} or damage zone ahead of the crack tip \cite{15,16}. “Dynamic Embrittlement” (DE) is often taken to imply that it is oxygen segregation in front of the crack that causes decohesion of grain boundaries. However “Stress Assisted Grain Boundary Oxidation” (SAGBO) suggests the formation of oxides which embrittle the grain boundaries. Recently, with advanced material characterisation and sample preparation techniques, such as atom probe tomography (APT), and focused ion beam for site-specific sample extraction prior to transmission electron microscopy (FIB-TEM), detailed chemical analysis at the crack tip is now possible. Recent observations address both oxidation and oxygen segregation at, and ahead of the crack tip \cite{17–19}.

Rather than the more familiar approach of employing relatively short dwell periods (up to 120 s), the present study adopts a long dwell period of 1 h to promote time dependent processes including grain boundary oxidation and matrix creep deformation. Here, the dwell fatigue crack growth of coarse grained RR1000 was investigated both in air and in vacuum. Together with the aid of TEM characterisation of crack tip oxides and observations of deformation, a novel mechanism for environmentally assisted crack growth is proposed.

2. Experimental

2.1. Material and heat treatment

The material examined in this article is RR1000, a powder processed, γ′ strengthened, nickel-based superalloy, developed for compressor or turbine discs of gas turbine aero-engines. The typical chemical composition of RR1000 is shown in Table 1.

The RR1000 material investigated here was processed through argon gas atomisation, powder sieving (≈270 mesh, \( \phi \approx 53 \mu \text{m} \)), hot isostatic pressing, hot extrusion and isothermal forging. All thermomechanical operations were conducted below the γ solvus temperature. A standard heat treatment procedure was then applied to all samples which were extracted from an as-forged pancake. This included a super-solvus solution annealing of 2 h at 1170 °C followed by an ageing heat treatment of 16 h at 760 °C. The super-solvus solution heat treatment produced a coarse grained microstructure due to complete dissolution of γ′ precipitates including incoherent γ′ particles (1–5 µm) that reside on grain boundaries. Two cooling rates (1.8 and 0.7 °C/s) were applied after solution annealing to simulate quenching conditions that could potentially be experienced in skin and rim (1.8 °C/s), and subsurface (0.7 °C/s) regions of disc forgings. These are referred to as FC and SC samples in the following text, respectively. The third heat treatment condition considered here included a high temperature stabilisation (HTS) anneal, 2 h at 857 °C, between the solution treatment and the final ageing treatment. A fast cooling rate from the solution heat treatment was used for these specimens. Note that these specimens were extracted from a different forging geometry than the one used for investigating the effects of cooling rate. This third set of samples is referred as HTS in the following text.

2.2. Microscopy and dwell fatigue crack growth tests

Microstructures and γ′ distributions resulting from the three heat treatments were studied using optical microscopy and scanning electron microscopy (SEM). Samples were mounted in conductive Bakelite and polished. They were initially etched with Kalling’s reagent (5 g CuCl₂ + 100 ml HCl + 100 ml ethanol) to reveal grain boundaries for grain size measurements. These samples were then re-polished and electrolytically etched in 10% phosphoric acid which removes the γ matrix and leaves γ′ in relief. High resolution images of γ′ particles were taken at a magnification range between 30 and 80k, using a Field Emission Gun (FEG) SEM. These images were then analysed using ImageJ software to obtain sizes and distributions of γ′ particles. At least three randomly selected images of appropriate magnifications were analysed to ensure the γ′ size-frequency histograms generated were representative. The γ′ distributions after mechanical testing were also examined in order to evaluate possible ageing effects during testing.

Dwell fatigue crack growth tests were conducted in air under constant amplitude loading with a load ratio of 0.1, using square cross-section (7 × 7 mm²), corner notched testpieces, subjected to uniaxial tension. The testpiece geometry adopted produces a crack profile of a quarter-circular shape under baseline fatigue loading. Tests were conducted using two alternating blocks of trapezoidal loading waveform: (1) with a long dwell period (3600 s) at peak load and (2) with a 1 s hold at peak load (baseline fatigue loading). The durations for load ramp-up, ramp-down and holding at the minimum load were all set at 1 s for both loading waveforms. The second loading waveform generates a baseline of fatigue crack growth rates, to which cyclic crack growth rates obtained from the first loading waveform can be compared. In addition, tests were also conducted under a vacuum of ~2 × 10⁻² mbar, in an attempt to isolate the potential contribution of creep alone from potential combined effects of creep and oxidation present in tests carried out in air. Temperatures ranged from 550 to 775 °C for vacuum tests but focussed on a single temperature of 700 °C for the tests conducted in air. A direct current potential difference (d.c.p.d.) technique was utilised to monitor crack growth continuously. Note that the alternate block loading approach employed here has several advantages. First, it can significantly reduce testing time, compared to growing the crack only under dwell fatigue cycles; second, it offers the opportunity to observe dwell
crack growth behaviour and baseline fatigue behaviour on the same testpiece (thereby minimising the potential influence of microstructural variations between testpieces), and can highlight any unusual (transient) crack growth behaviour above and below fatigue baseline behaviour on returning to fatigue baseline cycling following dwell cycles; and third, under intermittent baseline loading, the crack shape is found to return to near quarter-circular and this procedure prevents excessive crack tunnelling which is often observed in tests under long dwell cycling alone.

After testing, all fracture surfaces were examined using SEM. Particular care was taken to identify failure mechanisms, transgranular and/or intergranular crack growth and to observe any signs of creep deformation and damage.

In order to characterise oxidation ahead of the crack tip and to observe deformation mechanisms, a Quanta 3D FEG workstation which combines a focused ion beam microscope and scanning electron microscope (FIB–SEM), was used to prepare samples for transmission electron microscopy (TEM). FIB samples were extracted after fracture and from tests interrupted during stable crack growth so that crack tip oxidation and deformation mechanisms could be investigated under TEM. Analytical TEM studies were performed using a Philip TECNAI F20 microscope equipped with an Oxford Instruments silicon drift detector operating at an accelerating voltage of 200 kV. A Link Isis EDX (energy dispersive X-ray analysis) system was used to produce elemental maps.

3. Results and discussion

3.1. Microstructural variables

The application of a supersolvus solution heat treatment resulted in coarse grain microstructures as shown with an example of a FC sample in Fig. 1a. The grain size was observed to vary according to forging geometry and parameters. Typical grain size distributions are shown in the cumulative distribution function in Fig. 1b. The mean equivalent area grain diameter was 49 µm for the FC and SC sample (large pancake), and 17 µm for the HTS sample. The finer grain size in the HTS forging is anticipated to result from better uniformity of mechanical energy received during forging which encouraged even and numerous grain nucleation and growth. Grain boundary morphology, i.e., the degree of grain boundary serration, however, was in fact sensitive to the cooling rate. A comparison is provided in Fig. 1. The high magnification micrographs (Fig. 1c and d) clearly show much significant serration of grain boundaries associated with the slow cooling rate of 0.7 °C/s. An earlier study [20] suggested that such grain boundary serration results from protruding of growing secondary γ′

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Fig. 1. Typical grain microstructure under optical microscopy (example is for a FC sample) (a), and equivalent area diameter grain size versus estimated cumulative probability of occurrence for HTS, FC and SC samples (b). A comparison of grain boundary serration (secondary electron SEM micrographs) between FC (c), and SC (d).
particles at both sides of grain boundaries during cooling. Apart from the γ matrix and main strengthening phase γ', the material also contained a dispersion of sub-micron minor phases including MC carbides and M$_3$B$_2$ borides.

Two general size ranges of γ' precipitates, namely secondary and tertiary, result from the heat treatment procedures applied in the current study. In Fig. 2a and b, relatively low magnification micrographs illustrate the effect of the two cooling rates (1.8 and 0.7 °C/s) on the sizes and morphologies of the secondary and tertiary γ' precipitates. Such micrographs are sufficient to evaluate the distribution of secondary γ' precipitates, Fig. 2c. Although tertiary γ' precipitates can be seen in both Fig. 2a and b, higher magnification images are needed to establish the distribution of these much smaller γ' precipitates. Such micrographs are shown in Fig. 2d and e together with their distributions in Fig. 2f. Note that tertiary γ' is commonly defined based on size when performing image analysis, i.e., tertiary γ' ≤ 50 nm; secondary γ' > 50 nm. Here attempts are made to classify according to temperature and/or sequence of formation, noting the variation of precipitate chemistry. Some large γ' precipitates (>50 nm), which exist at the centre of channels between large and widely spaced secondary γ' particles, are classified as tertiary γ' in the current study.

The size, distribution and morphology of secondary γ' change significantly according to cooling rate. A uniform population of dense and spherical precipitates is observed with the faster cooling rate. The modal size is approximately 160 nm (Fig. 2c). Note that the size of the γ' precipitates is all given in terms of equivalent area diameter. The slower cooling rate resulted in a bi-modal distribution demonstrating the tendency to develop a population of large secondary γ' of complex morphology that appears to have multiple protrusions (modal size: ~360 nm) in addition to a second distribution of smaller spherical precipitates (modal size: ~150 nm), which is closely similar to the single distribution measured for the faster cooled sample. Such morphological instability is mainly driven by lattice mismatch between γ and γ' phases and has been investigated in CH98, Udimet 720Li and trial variants of RR1000 [21–23]. Although fewer in number, the larger precipitates seem to dominate in terms of area occupied as shown in Fig. 2b. It is also possible that some small precipitates seen on the metallographic images actually connect to the larger precipitates due to sectioning through protrusion tips of the large precipitates [24]. Hence, the actual population of smaller precipitates may have been enhanced in Fig. 2c.

The effect of cooling rate on the size of the smallest γ' is more subtle, but is still noticeable by carefully comparing Figs. 2a and b. Larger precipitates (~45–100 nm) are only observed in the slowly cooled sample to reside in the middle of the channels between secondary γ' precipitates, and characterised quantitatively by a tail deduced to have been added onto a distribution that is otherwise similar to that found in the FC sample, as shown in Fig. 2f. As expected all tertiary γ shows a spherical morphology.

The mean size of secondary and tertiary γ' particles has been measured to be 160 and 15 nm for the fast cooling rate of 1.8 °C/s, and 208 and 20 nm for the slow cooling rate of 0.7 °C/s respectively. The volume fraction of the secondary particles is approximately 40% for both cooling rates. The

Fig. 2. Secondary electron SEM images of γ' precipitates viewing at different magnifications for: (a) and (d) FC (1.8 °C/s), and (b) and (e) SC (0.7 °C/s). Comparisons of histograms of secondary γ' precipitates, (c), and tertiary γ' precipitates, (f), between FC and SC conditions.
volume fraction of tertiary $\gamma'$ is difficult to quantify, but it is considered that all $\gamma'$ will sum to a total volume fraction of $>45\%$ for the material investigated here [25]. After the standard solution and ageing heat treatment, tertiary $\gamma'$ precipitates are densely packed and uniformly distributed in the $\gamma$ matrix between secondary $\gamma'$ precipitates.

For the heat treatment with a 2 h stabilisation anneal at 857 °C (HTS) studied here, significant coarsening of the tertiary $\gamma'$ precipitates occurs. This is illustrated in Fig. 3a and c where the distribution (following this and ageing) is compared to that with the faster cooling rate (and ageing). The mean size of the tertiary $\gamma'$ precipitates is now 48 nm. However, the most striking feature with this HTS and aged condition is the scarce and non-uniform distribution of tertiary $\gamma'$ precipitates. As a result some areas of the channels between the secondary $\gamma'$ precipitates are free of tertiary $\gamma'$ precipitates. The secondary $\gamma'$ is also observed to increase in size which is manifested by a shift of the overall profile to the right (larger sizes) as shown in Fig. 3b. This observed distribution differs from the bi-modal distribution seen in the more slowly cooled sample, compare Fig. 3b to Fig. 2c. Although standard ageing has also been applied after HTS, it is considered that the microstructure is primarily set by the particular solution, quenching and HTS treatment.

3.2. Characterisation of different crack growth failure mechanisms

Due to the insertion of a holding period into the fatigue loading waveform, dwell fatigue crack growth at high temperature in air could potentially result from a combination of different failure processes including cyclic dependent fatigue, time dependent environmental embrittlement, and time dependent creep deformation and creep damage. It is important first to understand individual failure processes before considering their potential interactions. By changing the combination of loading waveform and environment, it is possible to observe and hence to highlight different failure processes.

3.2.1. Failure mechanisms in vacuum

Fatigue crack growth resistance ($da/dN$ vs $\Delta K$) without the involvement of time dependent effects was quantified by applying baseline fatigue loading (1 s dwell, 0.25 Hz) without the oxidising environment and with only a minimal (1 s) holding period at peak load, crack growth is deduced to result solely from a cycle dependent fatigue process. Indeed this is the case as verified by a flat transgranular fracture morphology at all test temperatures (Fig. 5a) including the highest temperature of 775 °C. Overlapping crack growth data suggest that fatigue crack growth resistance (in the Paris regime) is insensitive to temperature and microstructure ($\gamma'$ size and distribution) within this temperature range investigated in vacuum.

Dwell fatigue crack growth testing in vacuum is a technique often employed to identify potential creep damage in air as a vacuum environment minimises the contribution of...
oxidation and/or oxygen embrittlement [26]. Here with the application of a one hour dwell time the contribution of fatigue is also depressed and the potential for time-dependent creep damage is increased. Vacuum dwell crack growth resistance curves are given in Fig. 4b for the temperatures 600, 700, 750 and 775 °C. However, acceleration of crack growth rates from those of baseline fatigue is only measured at test temperatures of 750 °C and above, where a sudden increase of approximately two orders of magnitude is recorded. Fig. 5 compares fracture surface morphologies resulting from 1 s and 1 h dwell at the temperature of 750 °C. The predominantly transgranular morphology from the 1 s dwell (Fig. 5a) is replaced with a fully intergranular crack surface morphology for the 1 h dwell test. In addition the fracture surface is decorated by well developed microvoids (see Fig. 5b). Together with the observation of obvious bulk deformation of the testpieces (Fig. 6a), creep damage along grain boundaries and global (bulk) creep deformation within the matrix are deduced. It is expected that such a failure mode might occur and participate in the failure process in air at such temperatures. Although, in detail, the use of ΔK can be questioned when used to characterise crack growth resistance for a situation where bulk creep deformation dominates, it is clear from Fig. 4b that crack growth rates increase sharply in vacuum at the test temperatures of 750 and 775 °C. At lower temperatures, such as 700 °C, the crack growth rate for a FC testpiece featured a somewhat atypical sharp deceleration.

Fig. 3. Secondary electron SEM micrograph of γ precipitates after the HTS heat treatment (a), and the histograms of secondary γ precipitates (b), and tertiary γ precipitates (c), comparing to FC condition.

Fig. 4. Fatigue and creep crack growth resistance curves at temperatures 600, 700, 750 and 775 °C. Acceleration of crack growth rates from those of baseline fatigue is only measured at test temperatures of 750 °C and above, where a sudden increase of approximately two orders of magnitude is recorded.

Fig. 5. Fracture surface morphologies resulting from 1 s and 1 h dwell at the temperature of 750 °C. The predominantly transgranular morphology from the 1 s dwell (Fig. 5a) is replaced with a fully intergranular crack surface morphology for the 1 h dwell test.
in crack growth rates for a small increase in $\Delta K$ (Fig. 4b). Although the crack growth rates measured are of similar magnitude to the baseline fatigue crack growth rates, the mechanism is totally different as an intergranular fracture surface morphology is observed. Such crack growth is governed by time dependent localised creep deformation which leads to stress relaxation and crack blunting.

3.2.2. Failure mechanisms in air

From a comparison with the crack growth rates obtained in vacuum (700 °C), it can be inferred that any acceleration of crack growth rates in air at this temperature (for a similar microstructural condition) is due to the effect of the environment. Crack growth resistance curves for all three microstructural conditions are shown in Fig. 7. Note that different regions of crack growth from baseline fatigue block loading and 1 h dwell block loading are clearly visible on the fracture surface as shown in the example of Fig. 7b. Under high magnification the crack path under 1 h dwell block loading is fully intergranular for all cases (Fig. 8b), in contrast to the transgranular failure found in the baseline fatigue block loading regions (Fig. 8a). Although being predominantly intergranular, the fracture surface morphology seen in air (Fig. 8b) is also different to the ductile intergranular fracture surface seen in vacuum (Fig. 5b). There are no microvoids present on the grain boundaries, instead
the fracture surface is covered by oxides (Fig. 8c). In addition there is little sign of matrix deformation as the grain boundary ridges stay sharp and clear, which leads to a good retention of the original cross-section shape (Fig. 6b). It is open to question whether there is involvement of fatigue processes when the failure mode is fully intergranular although periodic unloading and reloading may enhance intergranular fracture and modify the crack tip stress field via reversed yielding.

3.3. Environmentally assisted dwell fatigue crack growth

3.3.1. Different crack growth behaviour

Distinct differences in crack growth resistance have been observed under 1 h dwell loading in air for the three different microstructures, as illustrated in Fig. 7. The testpiece that was fast cooled and aged (“FC” in Fig. 7a), demonstrated stable crack growth increments \( (da/dN) \) which relate to the stress intensity factor range \( (\Delta K) \) by the usual type of power law relationship (Paris), of a form similar to those seen in baseline fatigue crack growth resistance curves but with much increased crack growth rates \( (da/dN) \). However, for a slower cooled and aged testpiece (“SC” in Fig. 7a), decreases in dwell crack growth rates \( (da/dN) \) with crack advance were observed at lower \( \Delta K \) values (<25 MPa√m) which could potentially lead to arrest of the crack. Continued testing of “SC” at higher \( \Delta K \) values of 30 MPa√m and above after the first period of dwell testing at lower \( \Delta K \) values demonstrated again slower crack growth rates than the fast cooled specimen, but the differences were reduced. The best crack growth resistance is observed with HTS samples. A similar behaviour to SC was observed but with faster rate reductions in the low (<25 MPa√m) \( \Delta K \) region, and slightly slower crack growth rates in the high (>30 MPa√m) \( \Delta K \) region. Undoubtedly the microstructure (size and distribution of \( \gamma \) precipitates in the current study) has a profound influence on dwell fatigue crack growth resistance especially in low \( \Delta K \) regions (<25 MPa√m). Note that more than five tests were performed at each condition and the two sets of behaviour shown are representative of the general trends.

Thus a trend can be drawn which is that increased dwell crack growth resistance is associated with coarser and/or fewer \( \gamma \) precipitates. However, such a dependence seems to be much stronger for tertiary \( \gamma \). Fine secondary \( \gamma \) alone may not result in fast crack growth rates as for the case of HTS samples. On the contrary, coarser and fewer tertiary \( \gamma \) precipitates always led to improvement in dwell crack growth resistance even when there were fine secondary \( \gamma \) precipitates present. Fig. 9 shows the correlation between crack growth rates and the mean size of tertiary \( \gamma \). It seems to suggest that once a certain value is exceeded, slow crack growth rates may be obtained and they become less sensitive

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Fig. 5. Secondary electron SEM fractographs showing: (a) after baseline fatigue cycling; and (b) after 1 h dwell fatigue cycling, both at 750 °C in vacuum. Note that the crack growth direction is arrowed in each case.

Fig. 6. Secondary electron SEM fractographs showing overviews of the fracture surface after 1 h dwell testing at: (a) 750 °C in vacuum (FC); and (b) 700 °C in air (SC). Note that the crack originated from the top left corner in both testpieces.
to further size increase of tertiary $\gamma'$; such an average tertiary $\gamma'$ particle size is around $\sim$25–28 nm for the alloy investigated here. However, it should be stressed that the mechanism by which tertiary $\gamma'$ distributions affect dwell crack growth resistance is likely to be complex and may not be fully represented by mean size alone. Indeed, here it has been suggested that clear $\gamma$ channels free from tertiary $\gamma'$ precipitates may also play a role. Such an effect is consistent with the behaviour of the HTS specimen. By examining the microstructure after testing, it is believed that overageing had occurred for the SC sample during testing, especially in the first dwell block loading which lasted $\sim$233 h. A micrograph of the SC sample taken after testing (total testing time is 376 h) is shown in Fig. 10a. Overageing in this sample, compared to overageing in HTS (Fig. 3a), is subtle and only apparent for the tertiary $\gamma'$ distributions. A comparison between prior and post testing tertiary $\gamma'$ distribution is given in Fig. 10b. The $\gamma$ channels which were densely occupied by tertiary $\gamma'$ precipitates originally (Fig. 2e) become less densely populated. The broadening of the distribution after testing thus suggests coarsening as well as the potential dissolution of smaller tertiary $\gamma'$ precipitates.

3.3.2. Grain boundary embrittlement mechanisms

Two embrittlement mechanisms are considered in the literature to address environmentally related intergranular crack growth: one suggests stress accelerated grain boundary oxidation (SAGBO) [27], whereas the other claims dynamic embrittlement [28]. The two mechanisms are different in that weakening of grain boundaries is caused by the local formation and rupture of brittle oxides in the former case, but by decohesion of metallic bonds due to segregation of the embrittling oxygen species for the latter case. It appears to the present authors that any elemental oxygen will react with the major elements present in the alloy such as Ni, Co, Cr, Ti and Al due to the large negative free-energy change upon formation of their oxides. The affinity of Cr, and Al in particular, to oxygen has long been utilised as oxygen getter and provide high temperature oxidation resistance to superalloys. Indeed, the presence of oxide intrusions ahead of a fast moving crack has been confirmed for the same alloy at a temperature of 650 °C in previous work and has been reported recently [18].

In order to confirm the crack growth mechanism here and understand the observed behaviour, TEM–EDX...
characterisation has been performed in the vicinity of the crack tip of the interrupted testpieces. The TEM samples were produced by FIB milling. An overview of the interrupted cracks is shown in Fig. 11. The details of sample preparation and characterisation are similar to those described in [18]. Consistent with previous work [18], it is again confirmed that grain boundary oxide intrusions are always present ahead of the crack tip as well as in its wake. The oxide scale associated with the decelerating crack is significantly thicker than for the fast growing crack, especially in the wake of the crack tip, indicating a much extended period to grow the crack. In addition to the obviously different appearances, the types of oxide present at the furthest extent of the oxide intrusion, i.e. approximately a few microns ahead of the crack tip, were also different. Fig. 12 provides a comparison of constituents in the oxide intrusion ahead of a crack growing at a rate of approximately 36 \( \mu \text{m/h} \) in a FC testpiece, and a decelerated crack in a SC testpiece. In the case of the fast growing crack (FC), Co and Ni oxides are the main constituents present in the middle of the intrusion, and thin Cr oxides only exist between the main body of the oxide intrusion and the metal matrix. Associated with the crack that was growing at a much reduced rate of 1.5 \( \mu \text{m/h} \) in a SC sample, \( \text{Cr}_2\text{O}_3 \) and \( \text{Al}_2\text{O}_3 \) were the main constituents. Co and Ni oxides disappeared at the furthest extent of oxide intrusion. These observations are consistent with other studies [17–19], which support the argument that formation of oxides ahead of the crack tip and their rupture is the primary cause of environmentally assisted crack growth, a process captured by the term SAGBO in the literature.

### 3.3.3. Kinetics of intergranular oxidation ahead of crack tips

In order to understand how crack tip oxidation contributes to crack growth rate, the kinetics of oxidation (associated with a crack tip geometry) and the influence of external applied stress needs to be considered. It has been found that the crack tip oxide intrusion possesses a similar sequence of layering of different oxides as observed on the oxide scale formed on stress free flat coupons [17–19]. However, significant enhancements over the stress-free flat surface geometry were also observed both with and without the application of external stress. An enhancement factor \( S_r \), which is the ratio of the rate of growth of the oxide intrusion in the stressed state to that in the unstressed state at a given length of intrusion, is dictated by the stress normal to the direction of growth of the oxide intrusion, is derived elsewhere [29]:

\[
S_r = \exp \left( \frac{\sigma_k \Omega_d (\Phi - 1)}{kT} \right)
\]  

Fig. 8. Secondary electron SEM fractographs after testing at 700 °C in air: (a) in baseline fatigue region; (b) in 1 h dwell loading region; and (c) a high magnification image of (b) showing oxidised surface. Note that the transition from baseline fatigue region to 1 h dwell loading region is arrowed in (a), and the crack growth direction is from bottom left to top right in all cases.
where \( \sigma_{\text{tip}} \) is the stress at the tip of the intrusion, \( V_M \) is the volume of the metal atom in the alloy, \( \Phi \) is the ratio of oxide to metal volume, approximated here by the Pilling–Bedworth ratio, \( k \) is Boltzmann's constant and \( T \) is absolute temperature.

Limited oxidation experimental results are available in the literature for a crack tip geometry. Here we use the results from a previous study which considered the formation of oxide intrusions ahead of cracks in RR1000 of a different microstructure [18]. In [18], two cracks were exposed at 650 °C in air for 5 h without oxide cracking: one with applied stress the other without. The resulting oxide intrusions were observed to be chromia (~3 μm) and NiO (~9.7 μm) respectively. From these the rate constant at 650 °C, \( k_{p,650} \), can be calculated assuming parabolic growth kinetics, i.e., \( \ell = k_p t \) (where \( \ell \) is intrusion length and \( t \) is elapsed time). The length of chromia intrusion and NiO type oxide intrusion formed after 1 h at 700 °C under the same stress enhancement as in [18] can be estimated by using an Arrhenius activation energy on \( k_p \), \( Q \), which is 258,000 J mol\(^{-1}\) for chromia growth [30] and 170,000 J mol\(^{-1}\) for NiO growth [31].
where $R$ is the gas constant, 8.314 J mol$^{-1}$K$^{-1}$.

Values of 3.2 and 7.7 μm can be calculated for the length of the Cr$_2$O$_3$ and NiO intrusions, respectively. Note that these calculations consider the same stress enhancement as in [29], i.e., a crack-tip stress of 2.8 and 2.3 GPa. The stresses at the oxide intrusion tip in the present study, however, are expected to be lower due to the different microstructure and higher test temperature. Assuming a $\sigma_p$ of 2 GPa for both Cr$_2$O$_3$ and NiO intrusions, lower $S$ values of 7.0 and 2.9 (17.6 and 3.6 μm in [29]) can be calculated for Cr$_2$O$_3$ and NiO intrusions respectively. Taking this effect into account, a more realistic estimation of Cr$_2$O$_3$ and NiO intrusion length for the current study, after 1 h holding at the peak load at a temperature of 700 °C (provided that they do not crack), will be 1.3 μm and

$$k_{p-700°C} = k_{p-650°C} \times \exp \left( \frac{Q}{R} \left( \frac{1}{923} - \frac{1}{973} \right) \right)$$ (2)

Fig. 12. A STEM bright field image, and corresponding EDX maps showing oxides ahead of the FC (a), and SC (b), interrupted cracks. The relative concentration in the EDX maps increases from low to high in the sequence: black, blue, green, yellow and red. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
6.2 μm respectively. Note that the calculations use \( \Omega_{\text{Cr}} = 1.22 \times 10^{-29} \) and \( 1.10 \times 10^{-29} \) for Cr and Ni, and \( \Phi = 2.07 \) and 1.65 for \( \text{Cr}_2\text{O}_3 \) and NiO respectively. It should be mentioned that the stress state at the crack tip, in addition to enhancing oxidation, could alter the type of oxides that formed as seen in [18], and most importantly dictates the fracture of oxide intrusions which is discussed later.

It is encouraging to note that the estimated chromia intrusion length is similar to the growth rate at the interruption (1.5 μm/cycle) for the SC sample, and the actual intrusion length present at the crack tip (Fig. 11b). This supports the assumption that no cracking occurs during the 1 h dwell period for the decelerated crack in the SC sample. However, the oxide intrusion may not survive dynamic unloading and reloading at the end of the dwell period, as indicated by a step increase of potential drop (p.d.) across the crack face. It is understood that the fracture toughness of \( \text{Cr}_2\text{O}_3 \) and NiO at 700 °C is very low, \( \sim 2.0 \) MPa m/√m [32]. Actual stresses seen in oxide intrusions are complex and are likely to be affected by external mechanical stresses, volume expansion induced by oxide formation and crack morphology (blunting due to accumulation of creep deformation). Such understanding needs detailed stress analysis of the oxide intrusion and surrounding matrix which is under investigation currently. After cracking, the oxide intrusion re-grows during the next dwell period. This demonstrates intermittent oxide cracking and crack growth.

For the FC testpiece, the fact that the estimated NiO intrusion length is significantly longer than that seen at the crack tip (Fig. 11a) could simply suggest that cracking has already taken place before it can reach such a length during the 1 h dwell. Such cracking could plausibly start at an early stage of dwell period and may happen many times within the holding period. An extreme scenario and for the most susceptible microstructure the interval between oxide cracking could be reduced to such an extent that cracking occurs continuously. The presence of NiO and CoO at the very extent of the oxide intrusion supports such an argument, because by breaking the oxides at an early stage, the formation of protective chromia may be inhibited and an oxygen partial pressure higher than the dissociation oxygen partial pressure of NiO and CoO maintained. In this manner, fast crack growth rates are inevitable.

The two contrasting crack growth behaviours are schematically demonstrated in Fig. 13. It is noticed that similar crack growth rates were measured between the SC and FC testpieces at the beginning of the first dwell block loading (Fig. 7a). A transition of cracking behaviour from dynamic (continuous) to intermittent (upon stress unloading and reloading) is expected to have occurred associated with crack tip blunting of the original (sharp) fatigue pre-crack tip. It is intriguing that such a transition occurred in SC samples only. Due to a competition between oxidation damage and creep deformation, it is deduced that the faster accumulation of creep deformation in the SC sample blunts the crack tip, and reduces (and/or eliminates) tensile stresses in the oxide intrusion, both delay fracture of the oxide intrusion.

### 3.3.4. Effects of stress relaxation

There is a link between the size and distribution of \( \gamma' \) precipitates and creep rate. A considerable amount of discussion is available in the literature. In a study conducted by Locq et al. [33], three ageing heat treatments were applied to achieve fine and numerous, coarser and less numerous, and complete elimination of tertiary \( \gamma' \) populations respectively for a coarse grain NR3 alloy. The time to achieve 0.05% creep strain under a stress of 650 MPa at 700 °C has been found to be \( \sim 75 \) h for the microstructure that had fine and numerous tertiary \( \gamma' \) (\( \sim 27 \) nm) precipitates, but reduced to \( \sim 40 \) h and \( \sim 0.75 \) h for the microstructures having coarser and no tertiary \( \gamma' \) precipitates respectively. Such differences in creep rate are dictated by the important role of tertiary \( \gamma' \) in blocking dislocation movement within \( \gamma' \) channels which could influence how dislocations interact with secondary \( \gamma' \) precipitates. The dominant deformation mechanism is observed to be precipitate shearing in FC sample (Fig. 14a) and precipitate looping in SC sample (Fig. 14b) after dwell crack growth testing. The observations are consistent with TEM observations on samples after creep testing in Locq’s work [33]. Other research on the influence of microstructure includes work from Telesman et al. [10] and Unocic et al. [34] conducted on a similar alloy LSHR, and ME3/Rene 104 respectively.

The degree of crack tip creep deformation can also be inferred from the crack growth resistance curves considered here. As local creep deformation blunts the crack tip and causes the local crack tip stress field to relax [10,12], the effective local stress available for fatigue crack growth is lowered. This is demonstrated by a reduction in fatigue crack growth rates below those under baseline fatigue loading after a dwell block loading as can be seen for “SC” after the first dwell block loading, see Fig. 7a. A certain number of fatigue cycles and crack extension are deduced to be required for the crack to re-sharpen and overcome the influence of the prior stress field, before crack growth rates recover to those of baseline fatigue (as shown in Fig. 7a). The degree of stress relaxation can perhaps be estimated roughly by the magnitude of crack growth rate reduction from fatigue baseline rates. In general, a reverse relationship between the dwell crack growth rates and degree of stress relaxation is observed. For a fixed microstructure, stress relaxation is more pronounced at lower \( \Delta K \) values (“SC” and “HTC” in Fig. 7a). In contrast, no such effects are observed in FC samples.
3.3.5. Effects of overageing

Among the three original microstructures investigated here, optimised crack growth resistance is obtained by the HTS and aged condition (Fig. 3a). Indeed, sharper crack growth retardations at low $\Delta K$ values ($<25$ MPa m$^{-1/2}$) made it very difficult to grow the crack, and the material suffers very little accelerated growth under dwell fatigue conditions. It is deduced, based on the previous discussion, that such benefit comes from faster stress relaxation around the crack tip, which is associated with fewer and coarser tertiary $\gamma'$. Perhaps a larger impact is plausibly from the existence of many clear $\gamma$ channels – free of tertiary $\gamma'$. Indeed a significantly larger influence on creep from the absence of tertiary $\gamma'$ than from coarse tertiary $\gamma'$ is suggested by Locq et al. [33]. In addition to overageing induced by heat treatment, it is understood that it can occur during service and our recent studies suggest it can be enhanced under the high local stresses in the crack – tip deformation zone.

Due to the significant influence of overageing on dwell crack growth resistance, it can be inferred that the fast crack growth rates seen in the FC testpieces are unlikely to be encountered during service. Despite extremely fast crack growth rates measured under dwell fatigue loading, such crack growth can only be activated when a critical mechanical driving force ($\Delta K_{th}$) is met. Work has been undertaken to determine the $\Delta K_{th}$ values for a coarse grain RR1000 and a fine grain Udimet 720Li [35] and it is observed to be generally higher than those for trans-granular fatigue crack growth. The implication is that with the time spent on initiating and growing a crack at lower $\Delta K$ ($<\Delta K_{th}$), overageing would take place, and the early intergranular crack growth would be then dominated by deceleration and stress relaxation.

4. Conclusions

The use of block cycles in terms of alternating frequencies/waveforms and long (1 h) dwell times, has revealed some interesting characteristics of dwell fatigue crack growth behaviour:

(1) At a temperature of $700^\circ$C in air, crack growth resistance under 1 h peak load dwell fatigue loading is extremely sensitive to the size and distribution of tertiary $\gamma'$ precipitates. Up to two orders of magnitude difference in cyclic crack growth rates ($da/dN$) can result between different microstructures. Both thermal processing and in-service thermal exposure can impact on dwell crack growth resistance curves.

(2) For coarse grain RR1000, crack growth at high temperatures of up to $700^\circ$C in air under long dwell fatigue loading is dominated by time dependent failure processes of environmental embrittlement. Creep crack growth processes are observed to dominate only at very high temperatures of $\geq 750^\circ$C.

(3) The effect of environment on dwell crack growth is complex. The failure mechanism involves oxide formation ahead of the crack tip grain boundaries and its fracture. Although it has a similar physical process as proposed by the stress assisted grain boundary oxidation (SAGBO) concept, the mechanism described in the current study emphasises the significance of microstructure and matrix creep deformation on fracture of oxide intrusions ahead of the crack tip.

(4) For coarse grain RR1000, excellent dwell crack growth resistance can be achieved at $700^\circ$C in air by applying appropriate cooling rates from the supersolvus heat treatment temperature and/or by introducing a high temperature stabilisation heat treatment. The dwell crack growth resistance can also be improved by in-situ overageing during long term testing at a temperature of $700^\circ$C.

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