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Prediction of Crack Growth in a Nickel-Based Superalloy under Fatigue-Oxidation Conditions

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Abstract

Prediction of oxidation-assisted crack growth has been carried out for a nickel-based superalloy at elevated temperature based on finite element analyses of oxygen diffusion, coupled with viscoplastic deformation, near a fatigue crack tip. The material constitutive behaviour, implemented in the finite element code ABAQUS via a user-defined material subroutine (UMAT), was described by a unified viscoplastic model with non-linear kinematic and isotropic hardening rules. Diffusion of oxygen was assumed to be controlled by two parameters, the oxygen diffusivity and deformation-assisted oxygen mobility. Low frequencies and superimposed hold periods at peak loads significantly enhanced oxygen concentration near the crack tip. Evaluations of near-tip deformation and oxygen concentration were performed, which led to the construction of a failure envelop for crack growth based on the consideration of both oxygen concentration and accumulated inelastic strain near the crack tip. The failure envelop was then utilised to predict crack growth rates in a compact tension (CT) specimen under fatigue-oxidation conditions for selected loading ranges, frequencies and dwell periods. The predictions from the fatigue-oxidation failure envelop compared well with the experimental results for triangular and dwell loading waveforms, with marked improvements

achieved over those predicted from the viscoplastic model alone. The fatigue-oxidation predictions also agree well with the experimental results for slow-fast loading waveforms, but not for fast-slow waveforms where the effect of oxidation is much reduced.

Keywords: Finite element analysis; Oxygen diffusion; Accumulated inelastic strain; Failure envelop; Crack growth rate; Nickel-based alloys.

Nomenclature

f	fatigue loading frequency
$\dot{\epsilon}_p$	viscoplastic strain rate tensor
f_1	von Mises yield function
σ	stress tensor
$\alpha(\alpha_1, \alpha_2)$	kinematic hardening variable tensors
R	isotropic hardening variable
$\dot{p}$	accumulated inelastic strain rate
E, ν	Young's modulus and Poisson's ratio
k	initial size of the viscoplastic threshold surface
b	speed towards saturation for the isotropic hardening variable
Q	saturated value for the isotropic hardening variable
C_1, C_2	saturated values of the kinematic hardening variables
a_1, a_2	speed towards the saturation for the kinematic hardening variables
Z, n	viscous parameters
C	concentration of oxygen

t	time
∇	gradient
D, κ	oxygen diffusivity and mobility
P	hydrostatic stress
D_1	reference diffusivity
Q_1	activation energy
R_1	universal gas constant
T	absolute temperature
Ω	molar volume of oxygen.
D_0, κ_0	oxygen diffusivity and mobility at 650°C
W, H	width and height of the CT specimen
a	crack length
C_0	oxygen concentration on the crack surface
P	load
$K, \Delta K$	stress intensity factor and its range
R_0	load ratio
d^*	characteristic distance ahead of the crack tip

1. Introduction

The detrimental effects of environmental factors, in particular oxygen, on high-temperature fatigue crack growth resistance in nickel alloys have been clearly demonstrated by experimental results and well documented in the literature [1-11]. For polycrystalline nickel superalloys, environmental effects were frequently observed to modify the fracture morphology from transgranular to intergranular

during crack growth, as well as to increase the crack growth rates significantly for a given stress intensity factor [5, 6, 8, 9]. Considerable effort has been made to understand the crack tip oxidation mechanisms in order to provide a basis for the development of quantitative models that predict crack growth under operational temperatures and loading conditions. A number of mechanisms have been proposed including oxide formation (Ni, Nb, Al, Cr) [5, 8, 9], vacancy injection [1], gas bubble formation (CO or CO₂) [2] and releasing of embrittling alloy elements [3] at the grain boundaries. As a result, several life prediction models have been developed for nickel alloys based on different perspectives of the fatigue-oxidation interaction process, such as the oxide-depth based model [6], the oxide-passivation based model [4] and the multiplicative mechanical-environmental damage model [7].

Evidence of oxidation reaction with nickel alloys has been presented for smooth specimens with detectable oxide layers on the surface and at internal grain boundaries, a result of oxygen attack [2, 12, 13]. Oxidation has also been evidenced during crack propagation tests, with oxides clearly observed on crack surfaces [5, 8, 10, 11, 14]. Temperature is one of the major factors identified in the study of fatigue-oxidation interaction, and oxidation-assisted fatigue crack growth can be several orders faster with the increase of temperature due to the enhanced coupling of fatigue and oxidation [e.g., 6, 12, 14]. For nickel alloys, the majority studies were carried out between 500°C and 900°C, such as the study of creep-fatigue-oxidation microcrack propagation at 500°C for alloy MAR-M247 [7], fatigue crack growth at 550°C [6], 650°C [5, 6, 9] and 700°C [10, 11] for alloy 718, and stress-assisted oxidation kinetics at 600°C and 900°C [12] for Ni-20Cr alloys. As shown in [12], under applied external stress, the thickness of oxide layers increased significantly at 900°C when compared to that at 600°C. For alloy 718, the fatigue crack growth rate increased about two orders when the

temperature was raised from 550°C to 650°C at low frequencies where the oxidation-fatigue interaction prevails [6]. Similar behavior was also observed in [14] for the study of fatigue crack growth in nickel alloy RR1000 at 650°C and 725°C, where long dwell periods imposed at peak loads was shown to promote oxidation-fatigue interaction.

Alloy RR1000 is one of the new generation polycrystalline nickel alloys, developed at Rolls Royce through a powder metallurgy route, to meet the demands of increasing turbine entry temperatures and rotational speeds for the latest aero-engines [14-16]. The material has a γ - γ' system strengthened by Ni₃Al (γ') precipitates. Our recent studies [15] showed that crack growth rates of RR1000 alloy predicted from viscoplastic deformation follow the same trend as the experimental data, i.e., the increase of crack growth rate with the decrease of loading frequency and the increase of superposed dwell period, due to the enhanced crack tip deformation at lower frequencies and longer hold periods. The predictions agree well with the test data at frequencies $f > 0.1\text{Hz}$ and dwell periods $t < 10\text{s}$, while marked under-predictions are noted at frequencies $f < 0.01\text{Hz}$ and dwell periods $t > 100\text{s}$. As discussed in [15], the difference may be due to the neglect of oxidation effects at high temperature. For alloy RR1000, it was experimentally observed that oxidation modified the fracture morphology from transgranular to predominantly intergranular during crack growth, as well as to increase the growth rates significantly at a given stress intensity factor [14, 16-18]. The effect of oxidation on crack growth becomes more pronounced at low frequencies and long dwell periods.

Recently, it has been acknowledged [19-21] that oxidation-assisted crack propagation in nickel alloys is a result of oxygen diffusion into a local area with increasing tensile stress, such as crack tips. The oxygen element attacks the grain boundaries by lowering boundary cohesion and prompts accelerated intergranular cracking [19-21]. The diffusion process is facilitated by high tensile stresses

near the crack tip [12, 22], especially for superimposed dwell periods at the maximum load where the crack tip is fully open and experiences sustained high tensile stresses. Oxygen-diffusion-assisted crack growth in nickel superalloys, also termed as dynamic embrittlement [23, 24], has been supported, though indirectly, by experimental observation in Inconel 718 [19, 21]. Firstly, the individual grain boundary facets were found to be smooth on the submicrometer level, and oxides were only observed on the fracture surfaces where the crack front had passed. Secondly, it was found that the cracking process is extremely sensitive to rapid changes in oxygen pressure, where fracture could be turned on and off in less than 10s by alternately pumping oxygen from the chamber and re-admitting it. Oxidation kinetics for nickel alloys at 650°C is extremely slow [25, 26], and oxidation reaction does not have sufficient time to occur within the order of seconds. Dynamic embrittlement was introduced to define a time-dependent intergranular brittle cracking process in which the supply of embrittling elements is due to grain boundary diffusion into an area of increasing tensile stress ahead of a crack tip [19-24]. Here, stress may be regarded as a driving force to accelerate the diffusion of embrittling elements into the crack tip and promote crack propagation by weakening grain boundary cohesion, i.e., a dynamic process of embrittlement.

The objective of this work is to investigate the process of oxygen diffusion into a crack tip and its subsequent effects on crack growth, in order to quantify the contribution of oxygen environment to overall crack growth for alloy RR1000 at 650°C. A sequentially coupled deformation-diffusion analysis [21, 22, 27, 28] was adopted to account for the effects of oxygen diffusion and viscoplasticity on crack growth. The material constitutive behaviour, implemented in the finite element software ABAQUS [28] via a user-defined material subroutine (UMAT), was described by a unified viscoplastic model with non-linear kinematic and isotropic hardening rules [29]. Diffusion of oxygen into the fatigue crack tip was assumed to be controlled by two parameters, the oxygen diffusivity and

deformation-assisted oxygen mobility. Finite element analyses of a compact tension (CT) specimen were carried out to reveal crack-tip oxygen concentration for selected loading conditions. A failure envelop was constructed for crack growth based on the consideration of both oxygen concentration and accumulated inelastic strain near the crack tip. The failure envelop was then utilized to predict crack growth rates in a CT specimen under fatigue-oxidation conditions for selected loading ranges, frequencies, dwell periods and loading waveforms. The predicted results were compared with the experimental results [18] and those predicted from the viscoplastic model.

2. Viscoplastic Deformation

The viscoplastic deformation is described by the unified constitutive equations [30], where both isotropic (R) and kinematic (α) hardening variables are considered during the transient and saturated stages of cyclic response. A power relationship is adopted for the viscopotential and the viscoplastic strain rate tensor $\dot{\epsilon}_p$ is expressed as [30]:

$$\dot{\epsilon}_p = \left\langle \frac{f_1}{Z} \right\rangle^n \frac{\partial f_1}{\partial \sigma}, \quad (1)$$

where f_1 is the von Mises yield function, σ the stress tensor, and Z and n are material constants. The brackets imply that $\langle x \rangle = x$ for $x > 0$ and $\langle x \rangle = 0$ for $x \leq 0$. Here, the von Mises yield function f_1 can also be interpreted as viscoplastic threshold surface as the response is rate-independent within the yield limit.

The evolution of the kinematic stress tensor α and the isotropic stress R is described through the following rules [30]:

$$\begin{cases} \dot{\alpha} = \dot{\alpha}_1 + \dot{\alpha}_2 \\ \dot{\alpha}_1 = C_1(a_1 \dot{\epsilon}_p - \alpha_1 \dot{p}) \\ \dot{\alpha}_2 = C_2(a_2 \dot{\epsilon}_p - \alpha_2 \dot{p}) \end{cases} \quad \text{and} \quad \dot{R} = b(Q - R) \dot{p}, \quad (2)$$

where C_1 , a_1 , C_2 , a_2 , b and Q are six material and temperature dependent constants which determine the shape and amplitude of the stress–strain loops during the transient and saturated stage of cyclic response, and $\dot{p}$ is the accumulated inelastic strain rate defined by [30]:

$$\dot{p} = \left\langle \frac{f_1}{Z} \right\rangle^n = \sqrt{\frac{2}{3}} d\dot{\epsilon}_p : d\dot{\epsilon}_p. \quad (3)$$

The constitutive equations contain eleven material parameters, namely, E , ν , k , b , Q , C_1 , a_1 , C_2 , a_2 , Z and n . The kinematic hardening behaviour is described by C_1 , a_1 , C_2 and a_2 , where a_1 and a_2 are the saturated values of the kinematic hardening variables, and C_1 and C_2 indicate the speed with which the saturation is reached. The isotropic hardening is depicted by Q and b , where Q is the asymptotic value of the isotropic variable R at saturation and b indicates the speed towards the saturation. The initial size of the viscoplastic threshold surface is represented as k , E is the Young's modulus, ν is the Poisson's ratio, Z and n are viscous parameters.

Values of the model parameters were optimized using the uniaxial experimental data of Alloy RR1000 at 650°C [29, 31], and the calibrated model simulates well the viscoplastic constitutive behaviour of the alloy for a range of loading conditions. The above material model has been programmed into a user-defined material subroutine (UMAT), using a fully implicit integration and the Euler backward iteration algorithm [15], and implemented in the finite element software ABAQUS [28].

3. Deformation-Assisted Oxygen Diffusion

Here we are concerned with the diffusive transport of oxygen into the crack tip under the influence of a mechanical loading field. The isotropic mass transport of solute species can be modeled as [27, 32]:

$$\frac{\partial C}{\partial t} = \nabla(D\nabla C - \kappa DC\nabla P) \quad (4)$$

where C is the concentration of the solute, t the time, ∇ the gradient, D the oxygen diffusivity, κ the oxygen mobility (or pressure factor) and P the hydrostatic stress (pressure). In fact, the constitutive law (4) for the mass diffusion is an enhanced form of Fick's law, which has been modified to include the pressure (equivalent to dilatational strain) gradient effects. The equation (4) was derived from a thermodynamics point of view, in terms of chemical potential of diffusive species [32], which has been generally used to consider the influence of mechanical stress on mass transport [e.g., 20, 22, 33-34]. The gradient of hydrostatic stress (or pressure) is considered to be the driving force for stress-assisted diffusion process.

The diffusivity D and mobility κ are material parameters which depend on temperature. The temperature-dependent relations for D and κ can be expressed as [27]:

$$D = D_1 \exp\left(-\frac{Q_1}{R_1 T}\right) \text{ and } \kappa = \frac{\Omega}{R_1 T} \quad (5)$$

where D_1 is a reference diffusivity, Q_1 the activation energy, R_1 the universal gas constant, T the absolute temperature and Ω the molar volume of oxygen.

Direct experimental measurements of the diffusivity parameters D_1 and Q_1 are very difficult and no definitive data available in the literature. Here the values were taken from those for Inconel 718 [27], with $D_1 = 10^{-4} \text{ m}^2/\text{s}$ and $Q_1 = 168.5 \text{ kJ/mole}$. From Eq. (5), the oxygen diffusivity was calculated to

be $D_0 = 2.9134 \times 10^{-8} \text{ mm}^2/\text{s}$ while the oxygen mobility was taken as $\kappa_0 = 0.1303 \text{ mm}^2/\text{N}$, where D_0 and κ_0 are the oxygen diffusivity and mobility at 650°C and assumed to be independent of the oxygen concentration. In the present work, no attempt was made to model chemical reaction kinetics, vacancy formation or the transport of other species [26] other than oxygen diffusion near the crack tip.

4. Finite Element Procedure

A standard CT specimen (width $W = 26 \text{ mm}$ and height $H = 15.6 \text{ mm}$) was considered and the finite element mesh for the specimen, as shown in Fig. 1, consists of four-noded, first-order elements with full integration [28]. Due to symmetry, only half of the specimen was considered. The element size in the crack tip/growth area is $12.7 \text{ }\mu\text{m}$, about twice the average grain size ($5\sim 6 \text{ }\mu\text{m}$) of the material. For crack growth study, it was recommended that the element size should be fine enough to capture the cyclic (reverse) plastic zone so that crack tip plasticity can be adequately evaluated [35]. In the present work, for $\Delta K = 20 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0.1$, the cyclic plastic zone is about 0.05 mm , which would contain approximately 5 elements (~ 10 grains); while for $\Delta K = 40 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0.1$, the cyclic plastic zone is about 0.18 mm , which would contain approximately 14 elements (~ 30 grains) [36]. Cyclic load was applied to a rigid pin fitted into the hole of the specimen. The rigid pin and the hole of specimen were treated as a pair of contact surfaces in ABAQUS [28]. For consistency, the same mesh was used for oxygen diffusion analyses. The crack length was chosen to be $a = 13 \text{ mm}$, i.e., $a/W = 0.5$.

To solve the overall fatigue-oxidation problem, a sequentially coupled mechanical-diffusion analysis was performed to include the dependence of oxygen diffusion on the stress state. The mechanical analyses were carried out first, with the history of hydrostatic stress for each integration point stored

in the ABAQUS output files. During the diffusion analysis, the output files were then used as inputs to include the effect of hydrostatic stress gradient on oxygen diffusion. Although the availability of oxygen may vary in the vicinity of the crack tip, as does the oxygen partial pressure, the oxygen concentration on the crack surface was assumed to be constant with a surface equilibrium value C_0 . A normalized measure for oxygen concentration (C/C_0) was adopted, in which the oxygen concentration of the virgin material is defined to be zero. The analyses were conducted at a constant temperature 650°C, and for load values of $P = 6.51$ and 8.68 kN, which corresponds to stress intensity factors of $K = 30$ and 40 MPam^{1/2}, respectively, for the CT specimen.

The mechanical deformation and oxygen diffusion are formulated at continuum level, where the material was treated as isotropic and homogeneous. Primary pathways for oxygen diffusion along grain boundaries and slip bands require studies at grain level using crystal plasticity coupled with oxygen diffusion, which is beyond the scope of the present work.

5. Near-Tip Oxygen Diffusion

Fig. 2(a) shows the contour plot of the normalised oxygen concentration near a fatigue crack tip after 10 loading cycles, where the stress intensity factor range $\Delta K = 30$ MPam^{1/2}, load ratio $R_0 = 0$ and loading frequency $f = 0.05$ Hz. The result shows the penetration of oxygen into the crack tip, as well as the crack flanks, due to oxygen diffusion process. It is also noted that oxygen concentration tends to accumulate at a location slightly away from the crack tip and off the crack growth plane, a consequence of load reversal. Fig. 2(b) shows the contour plot of the normalised oxygen concentration after 10 cycles for the 10s dwell loading situation, where the dwell is superimposed at peak loads of a 0.5Hz base line fatigue waveform. For this case, oxygen concentration is highly accumulated at the very tip of the crack. For comparison, Fig. 2(c) shows the contour plot for the

natural diffusion, i.e. without external load, where the duration for the natural diffusion was equal to 10 cycles for the 10s-dwell loading case. It can be seen that natural diffusion gives a uniform distribution of oxygen concentration with limited penetration. These results suggest that oxygen penetration is a process localised to the fatigue crack tip and greatly facilitated by the external load. Crack growth seems most sensitive to oxidation for dwell loading condition, as the crack remains fully open and experiences the highest stress level during the hold duration at peak loads.

Finite element analyses were also carried out for selected loading frequencies and superimposed dwell periods at peak loads (base line frequency = 0.5 Hz), and the results are shown in Figs. 3 and 4, respectively, after 10 loading cycles under $\Delta K = 30 \text{ MPam}^{1/2}$ and load ratio $R_0 = 0$. It is noted that oxygen diffusion was significantly enhanced with the decrease of loading frequency and the increase of hold periods, a result of prolonged exposure for oxidation. This indicates that the detrimental effects of oxidation on crack growth become more severe at lower frequencies and longer dwell periods, as manifested in the accelerated crack growth observed experimentally. It may also explain the under-predicted crack rates from the visoplastic analysis for low frequencies ($f < 0.01\text{Hz}$) and long dwell periods ($t > 100\text{s}$) [15]. Thus, the effects of oxidation on crack growth may be necessarily accounted for by the consideration of near-tip oxygen diffusion process, as further explored in the following section.

6. Failure Envelop for Crack Growth under Fatigue-Oxidation

In order to predict the crack growth rates under fatigue-oxidation conditions, it is necessary to formulate an appropriate criterion to account for the contributions from both mechanical deformation and oxidation. This requires necessarily parameters which represent the viscoplastic deformation and the oxidation effect. For nickel alloys at 650°C, the deformation-controlled crack

growth is a ductile fracture process, hence a strain-based parameter may be suitable to represent the contribution from mechanical deformation. For oxidation influence, the oxygen diffusion near the crack tip is assumed to be the controlling mechanism, and the normalised oxygen concentration is used here to represent the contribution from oxidation.

The idea is to use the two parameters, accounting for viscoplastic deformation and oxygen diffusion, to construct a failure envelop from which crack growth prediction under fatigue-oxidation may be made. Experimental crack growth data at selected testing conditions [18] were utilised to determine the number of cycles required to grow the crack in the FE model (Fig. 1). Specifically, the number of cycles was calculated by dividing the near-tip element size ($12.7\mu\text{m}$) with the measured crack growth rate. FE analyses were then carried out for the calculated number of cycles, i.e., just before the crack grows into the next element, and the values of the two parameters were extracted. The extracted values of the two parameters were then plotted against each other to obtain a failure curve. Ideally, if the two parameters are appropriate, the data points should collapse into a single curve, which can then be regarded as the failure envelop.

In the present work, the construction of fatigue-oxidation failure envelop was focused on choosing the appropriate parameter to represent the mechanical deformation, whilst the oxidation effect was represented by oxygen concentration. The crack growth data for selected loading frequencies were considered with a load ratio $R_0 = 0.1$ and $\Delta K = 30$ and $40 \text{ MPa}\sqrt{\text{m}}$. After careful evaluation, it was found that a combination of the accumulated plastic strain and normalised oxygen concentration is able to bring the data together. The results are presented in Fig. 5, where all the data seem to collapse into the fitted failure curve. Other strain-based parameters, such as the total strain, the plastic strain and the effective plastic strain, were found unsuitable, as they produced large scatters and were

unable to produce a single failure curve. The choice of the accumulated plastic strain seems justified in that, under fatigue loadings, the reversed deformation during unloading is accounted for as well as that during loading. A contour plot of accumulated inelastic strain near a fatigue crack tip after 10 loading cycles is shown in Fig. 6 for $\Delta K = 30 \text{ MPam}^{1/2}$, load ratio $R_0 = 0$ and loading frequency $f = 0.05\text{Hz}$, where the accumulated inelastic deformation is localized in the vicinity of the crack tip.

As shown in Fig. 5, three regions are identified for the failure envelop. In region I, the loading frequency is above 2.5Hz and crack growth is mainly controlled by mechanical deformation. While in region III, the loading frequency is below 0.001Hz and crack growth is mainly controlled by oxidation. Region II represents the transition in between, where crack growth is determined by the combined effects of the accumulated inelastic strain and the oxygen concentration. The three distinctive regions seem to be sufficient to cover the range of mechanisms for crack growth under fatigue-oxidation conditions. Outside the failure envelop, crack growth is assured due to various mechanisms.

Crack growth criterion proposed in the current work is essentially a strain-based approach, which has been widely used for the prediction of viscoplastic failures such as creep [e.g., 37-39]. The strains local to a crack tip are of multiaxial nature such that an accumulated strain, which accounts for all strain components, is often adopted as a damage parameter [e.g., 37-40]. This parameter is also used in the current work, where the progressive nature of the accumulative plastic strain with cycles, as opposed to the relative constant stress response, is considered to be the main crack driving force [15], and as an alternative to other criteria such as plastic work or J-integral approaches. In what follows, the constructed failure envelop in Fig.5 was utilised to predict the crack growth rates for selected loading ranges, frequencies, dwell periods and waveforms.

7. Predictions of Crack Growth Rates

Using the failure envelop constructed above, crack growth rates in a CT specimen at 650°C were predicted from the finite element analyses of crack tip deformation for selected loading conditions, for which the experimental results [18] were used for comparison. During the analyses, the accumulated inelastic strain and the oxygen concentration over the characteristic distance $d^* = 12.7\mu\text{m}$ [15] ahead of the crack tip were recorded against the number of cycles. The results were then used to obtain the number of cycles for the crack to grow, i.e., when the combination of the accumulated inelastic strain and the normalized oxygen concentration reached the failure envelop. The average crack growth rate was then calculated by dividing the characteristic distance with the number of cycles.

7.1. Effects of loading frequency

The predicted effects of loading frequency on crack growth rates are shown in Figs. 7 and 8 for $\Delta K = 30$ and $40 \text{ MPa}\sqrt{\text{m}}$, respectively, under a triangular waveform (load ratio $R_0 = 0.1$). Test data for CT specimens at 650°C [18] were also included for comparison. The predictions from the accumulated inelastic strain agree well with the test data for high frequency region ($f > 0.1\text{Hz}$), while marked difference is noted in the low frequency region ($f < 0.01\text{Hz}$). This is similar to those predictions in [15], where only viscoplastic deformation was considered. For the predictions based on fatigue-oxidation failure envelop, marked improvements were achieved for all loading frequencies.

7.2. Effects of dwell period

The effects of superposed dwell at peak loads on crack growth are predicted for $\Delta K = 30$ and $40 \text{ MPa}\sqrt{\text{m}}$, respectively, by considering a trapezoidal loading waveform with a baseline frequency $f = 0.25$, a load ratio $R_0 = 0.1$ and selected dwell periods. The predictions are presented in Figs. 9 and 10, including the experimental data for CT specimens at 650°C under the same loading conditions [18]. The predictions from accumulated inelastic strain show increased deviations from the experimental data as the dwell period is increased, especially when a dwell period is over 100 seconds. Again, this difference is drastically reduced when the fatigue-oxidation failure envelop was used for the prediction. The fatigue-oxidation predictions are very close to the experimental results for both short and long dwell periods. The predicted crack growth rates for 300s and 1000s dwell periods are from the oxygen concentration criterion ($C/C_0 = 4.54$), i.e., the region III of the failure envelop in Fig. 5.

7.3. Effects of loading waveform

The effects of loading waveform on crack growth rates are predicted by considering slow-fast case for $\Delta K = 30$ and $40 \text{ MPa}\sqrt{\text{m}}$, respectively. The predictions are presented in Figs. 11 and 12, including the experimental data for CT specimens at 650°C under the same loading conditions [18]. For slow-fast waveforms, the predictions from the accumulated inelastic strain show marked differences from the test data, which are significantly improved by using the fatigue-oxidation failure envelop.

In addition, crack growth rates for fast-slow waveforms are also predicted for $\Delta K = 30$ and shown in Fig. 13, where the experimental data for CT specimens at 650°C under the same loading conditions [18] were included for comparison. Interestingly, for fast-slow waveform, the predictions from the

accumulated inelastic strain agree with the experimental data very well, while the inclusion of oxygen diffusion led to severe over-predictions.

8. Discussions

For polycrystalline nickel alloys under low cycle fatigue at elevated temperature above 500°C, a transition from cycle-dependent transgranular crack propagation to time-dependent intergranular crack propagation may occur when the frequency is sufficiently low and/or the hold period at peak loads is sufficiently long, resulting in significant increase in crack growth rates. As frequency or cycle duration does not seem to influence the crack propagation rate at low temperatures or under vacuum conditions, oxygen from the environment may be the key factor for the time-dependent crack propagation behaviour, which is attributed to the oxidation reaction at grain boundaries in most studies. For instance, Miller et al. [10, 11] proposed that the formation of thin intergranular Nb_2O_5 films, which fracture in a brittle manner, leads to high crack propagation rates. This was also supported by studies on Ni-18Cr-18Fe and powder-metallurgical superalloys with varying Nb concentrations, where the material's sensitivity to environmental attack depends solely on the Nb content. On the other hand, strong environmental effects on fatigue crack propagation were also observed by Molins et al. [9] in Ni-Cr model alloys without any Nb. Molins et al. [9] proposed that epitactic NiO-formation ahead of the crack tip introduced the vacancies into near-tip materials, which act as nucleation sites for pores and microcracks and thereby give rise to high crack propagation rates. High Cr contents were suggested to bring beneficiary effects due to the much slower kinetics of Cr_2O_3 formation than that of NiO formation, and, in addition, Cr could scavenge O_2 near the crack tip and reduces the level of elemental O required for oxidation reaction [9].

The oxidation-reaction mechanism has been challenged by the dynamic embrittlement hypothesis in recent studies of crack growth in nickel alloys under fatigue-oxidation conditions [19, 21]. Dynamic embrittlement was introduced to define a time-dependent intergranular brittle cracking process in which the supply of embrittling elements is due to grain boundary diffusion into an area of increasing tensile stress ahead of a crack tip [23, 24]. The embrittling elements lower the cohesion of the grain boundary and facilitate crack advance. The case of hydrogen embrittlement of steels is one of the most important damage mechanism that fits in the dynamic embrittlement scheme [41]. Other established dynamic embrittlement cases include sulfur-induced stress relief cracking in steels [24], S-induced cracking in Cu-Cr alloys [42], O-induced cracking in Cu-Be alloys [43] and Ni₃Al [23], and Sn-induced cracking in Cu-Sn model alloys [24, 44].

Dynamic embrittlement for nickel superalloys has been supported by experimental observations in Inconel 718 four-point bending tests [19-21]. Firstly, the individual grain boundary facets were found to be smooth on the sub-micrometer level, and oxidation of NbC precipitates was only observed on the fractured surface where the crack front had passed. Secondly, the cracking process was found to be extremely sensitive to rapid changes in oxygen pressure. Crack growth could be turned off and on in less than 10s by alternately pumping oxygen from the chamber and re-admitting it. Given the fact that oxidation kinetics for nickel alloys at 650C is extremely slow [25], there is insufficient time for oxide formation to happen ahead of the crack tip within the order of seconds. Direct observations of internal oxidation and associated damage were only available on smooth specimens, which have been exposed to oxidizing environment for a considerably long time, mostly greater than 20 hours [2, 12, 13, 26]. The current work also showed that crack growth rates in air at high-temperature can be predicted well from the dynamic embrittlement hypothesis, though collecting experimental evidence of crack tip oxygen diffusion process and measurements of oxygen concentration near the crack tip

are challenging tasks and yet to be accomplished. In the present work, oxygen diffusivity was taken as that for Inconel 718 [27], therefore the predicted oxygen diffusion should be considered to be approximate. Nevertheless FE analyses were carried out by varying the values of the diffusivity and mobility parameters. Although the magnitude of the oxygen concentration varies as a result, the shape of the failure envelop remains almost unchanged. Also, as long as the same parameter values are used throughout, the predicted crack growth rates remain the same.

The failure envelop was constructed entirely from our FE analyses by considering both oxygen concentration and accumulated inelastic strain near the crack tip. This paper is a first attempt to apply the “dynamic embrittlement” approach for the predictions of crack growth in nickel alloys under fatigue-oxidation conditions, where FE analyses of oxygen diffusion were coupled with the viscoplastic analyses. Construction of the failure envelop was based on the experimental crack growth data, which were obtained from mechanical testing in air that would include both mechanical deformation and oxygen diffusion near the crack tip. The failure envelop was then applied to independently predict the crack growth rates for dwell loading conditions and slow-fast loading waveforms. A simpler empirical approach considering nonlinear oxidation kinetics might also fit some of the data, but our approach, stemming from fundamental mechanistic considerations of viscoplasticity and oxygen diffusion, seems more likely to produce more generic results hence wider application potentials. Admittedly, a uniform oxygen concentration ($C=C_0$) was assumed at all locations on the crack surface, which may dictate the shape of the failure envelop. A variable distribution of oxygen concentration near the crack tip may alter the shape of the failure envelop, which needs to be investigated further.

The construction of failure envelop and associated crack growth predictions are based on the values of the accumulated inelastic strain and the normalized oxygen concentration averaged over the element just ahead the crack tip, which corresponds to a characteristic distance of 12.7 μm ahead of the crack tip. The need to specify a critical distance in association with a fracture criterion is well established [37, 45-53], particularly for sharp cracks, where, due to the stress and strain singularities, numerical results are non-unique unless a critical distance is specified. McClintock [45, 46] developed a mechanistic analysis of crack extension by fatigue, employing a failure criterion based on plastic strain accumulation over a “structural size” of the material. Quantitatively, a structural size of 5 μm seems to fit well with the experimental results for three types of materials [46]. Krafft [47] proposed that ductile fracture would occur when the instability strain for a uniaxial tensile specimen is achieved at the crack tip over a distance equal to the spacing of void nucleating particles. Ritchie et al. [48] successfully predicted fracture when the critical stress was achieved over two grain sizes. Rawal and Gurland [49] reported a critical stress was achieved over a critical distance about 1.3 grain-sizes. Chen et al. [51] reported a characteristic distance of 13 μm for crack tip fracture in a C-Mn steel, over which the critical strain, the critical stress and the critical stress triaxiality criteria are satisfied simultaneously. Said and Tasgetiren [52] used the characteristic distance of 0.8~1.37 mean grain size to determine the static and dynamic fracture toughness for bcc metals and alloys using the critical stress criterion. In the present work, the chosen characteristic distance 12.7 μm is on the similar order to the others [45-49, 51, 52] and it is about twice the average grain size, where crack growth is assumed to be associated with the cracking of a couple of grains. Mesh sensitivity study showed that the strain distribution ahead of the crack tip has reached a good convergence at $d^* = 12.7\mu\text{m}$ for the mesh used in the present work (within 5% difference between the present mesh and a doubly refined mesh).

Tests showed that crack growth under slow-fast loading waveform is significantly faster than that under fast-slow waveform [18]. Plasticity-induced crack closure in fatigue crack growth in CT specimens was found to be very small, if any [36], and this is independent of the material constitutive behaviour. Hence the current analysis has not taken closure into account. This is reasonable as we observe that crack flanks (in the FE models) remain open during the entire loading/unloading periods. Were closure to play a part, the effect on the crack growth rate should be more or less the same for slow/fast and fast/slow, as it would affect both loading and unloading parts. The sharp difference between the two can only be explained when oxidation effect is considered.

Interestingly, as shown in Fig. 13, inclusion of oxygen diffusion leads to over-prediction of the crack growth rates under fast-slow loading waveform. This indicates that the loading and unloading parts may play different roles in the oxygen diffusion process. Molins et al. [9] studied the oxidation assisted fatigue crack growth in nickel based superalloys by varying oxygen partial pressure during a loading cycle. They found that oxidation effects occur primarily during the loading part of the cycle and part (about 10%) of the unloading cycle when the crack tip is in traction. Consequently, an oxidation sensitive time was proposed by Tong et al. [14] and correlated well with crack growth data, where only 10% of the unloading duration was accounted for. Therefore, oxygen diffusion may be significant during the entire loading period, while only partially useful during unloading, which may explain the much-reduced oxidation influence for fast-slow loading waveforms. The present work only considered the stress-gradient assisted oxygen diffusion, while neglecting the influence of loading or unloading waveform. Further investigations are required to improve the model, which can nevertheless predict most of the loading cases with the exception of fast-slow waveform. For the latter case, modification of the oxygen boundary conditions ($C=C_0$) and/or the diffusion law may be necessary, and this is being considered in our ongoing work.

9. Conclusions

Finite element analyses of a compact tension (CT) specimen were carried out to predict the oxidation-assisted crack growth for a nickel-based superalloy at elevated temperature. The oxidation effects were accounted for by considering the oxygen diffusion process near a fatigue crack tip. Crack-tip oxygen diffusion depends largely on the loading conditions, where low frequencies and superimposed hold periods at peak loads significantly enhanced oxygen concentration near the crack tip.

A failure envelop was constructed based on the consideration of oxygen concentration and accumulated inelastic strain near the crack tip. This envelop defines the onset for crack growth under fatigue-oxidation. Predictions of crack growth rates from this fatigue-oxidation failure envelop were conducted for selected loading ranges, frequencies and dwell periods, which compares well with the experimental results. The fatigue-oxidation predictions also agree well with the experimental results for slow-fast waveforms, but not for fast-slow waveforms, possibly due to the much reduced loading duration where the effect of oxidation seems to be most active.

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Fig.1 (a) Finite element mesh for a CT specimen geometry and (b) the refined mesh for crack growth area (the smallest size $\Delta = 12.7\mu\text{m}$).

Fig. 2(a) Contour plot of the normalized oxygen concentration C/C_0 near a crack tip after 10 loading cycles for a triangular waveform with $\Delta K = 30 \text{ MPa}\sqrt{\text{m}}$, frequency $f = 0.05 \text{ Hz}$ and load ratio $R_0 = 0$ at 650°C .

Fig. 2(b) A contour plot of the normalized oxygen concentration C/C_0 near a crack tip after 10 loading cycles for a 10s-dwell waveform with $\Delta K = 30 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0$ at 650°C.

Fig. 2(c) A contour plot of the normalized oxygen concentration C/C_0 near a crack tip for natural diffusion (without external load), where the duration is equivalent to 10 loading cycles for the 10s dwell waveform.

Fig. 3 Normalized oxygen concentration ahead of the crack tip for selected loading frequencies after 10 loading cycles for a triangular waveform with $\Delta K = 30 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0$ at 650°C .

Fig. 4 Normalized oxygen concentration ahead of the crack tip for different dwell periods after 10 loading cycles for a trapezoidal waveform with $\Delta K = 30 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0$ at 650°C .

Fig. 5 Failure curve expressed in terms of the accumulated plastic strain and the normalised oxygen concentration; the experimental results were from Dalby and Tong [18] for selected loading frequencies at $\Delta K = 30 \text{ \& } 40 \text{ MPa}\sqrt{\text{m}}$ and $R_0 = 0.1$.

Fig. 6 Contour plot of the accumulated inelastic strain near a crack tip after 10 loading cycles for a triangular waveform with $\Delta K = 30 \text{ MPa}\sqrt{\text{m}}$, frequency $f = 0.05 \text{ Hz}$ and load ratio $R_0 = 0$ at 650°C .

Fig. 7 The effects of loading frequency on crack growth rate for a triangular waveform with $\Delta K = 30 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0.1$ at 650°C ; comparison of the model prediction and the experimental results [18].

Fig. 8 The effects of loading frequency on crack growth rate for a triangular waveform with $\Delta K = 40 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0.1$ at 650°C ; comparison of the model prediction and the experimental results [18].

Fig. 9 The effects of dwell periods on crack growth rate for a trapezoidal loading waveform with $\Delta K = 30 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0.1$ at 650°C ; comparison of the model prediction and the experimental results [18].

Fig. 10 The effects of dwell periods on crack growth rate for a trapezoidal loading waveform with $\Delta K = 40 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0.1$ at 650°C ; comparison of the model prediction and the experimental results [18].

Fig. 11 The effects of slow-fast loading waveform (triangular) on crack growth rate for $\Delta K = 30 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0.1$ at 650°C ; comparison of the model prediction and the experimental results [18].

Fig. 12 The effects of slow-fast loading waveform (triangular) on crack growth rate for $\Delta K = 40 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0.1$ at 650°C ; comparison of the model prediction and the experimental results [18].

Fig. 13 The effects of fast-slow loading waveform (triangular) on crack growth rate for $\Delta K = 30 \text{ MPa}\sqrt{\text{m}}$ and load ratio $R_0 = 0.1$ at 650°C ; comparison of the model prediction and the experimental results [18].