A coupled kinetic-constitutive approach to the study of high temperature crack initiation in single crystal nickel-base superalloys

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Abstract

A rate-dependent crystallographic constitutive theory coupled with a mass diffusion model has been used to study crack initiation in single crystal nickel-base superalloys, exposed to an oxidising environment and subjected to mechanical loading. The time to crack initiation under constant load has been predicted using a strain-based failure criterion. A notched compact tension (CT) specimen containing a single casting defect, idealised as a cylindrical void close to the notch surface, has been studied. Finite element analysis of the CT specimen revealed that, due to the strong localisation of inelastic strain at the void, a microcrack will initiate in the vicinity of the void rather than at the notch surface. The numerical results have also shown that the time to crack initiation depends strongly on the void location. The coupled diffusion–deformation studies have revealed that environmental effects reduce the time to crack initiation due to the oxidation-induced material softening in the vicinity of the notch and void. The applicability of a failure assessment approach, based on the linear elastic stress intensity factor, $K$, to predict the crack initiation time under creep loading is examined and a probabilistic framework for prediction of component lifetime is proposed.

Keywords: Nickel superalloy; Fracture; Diffusion; Surface; Creep; Finite elements

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1. Introduction

Nickel base superalloys are widely used in aerospace and power generation applications, due to their excellent creep and fatigue properties at high temperatures. Such alloys contain a high volume fraction (typically 68%) of 0.5–1 μm size γ' (primarily NiAl) precipitates embedded in a nickel-based solid solution (γ) matrix. In this work, the single crystal nickel superalloy, CMSX4, is studied. Fig. 1 shows typical heterogeneities in the alloy at both the mesoscopic and microscopic levels. The mesoscale heterogeneities (Fig. 1a) are due to the presence of approximately 10–20 μm casting pores, usually found in the interdendritic regions, while the microscale heterogeneities (see Fig. 1b) result from the 0.5–1 μm γ' precipitates. Since the thickness of a typical component (e.g. gas turbine blade) is on the order of millimetres, the component life is generally dominated by short crack initiation and growth. Observations of fracture surfaces in nickel superalloy creep specimens, loaded in a (001) crystallographic direction, have revealed multiple (001) facets, resulting from the propagation of microcracks, which appear to have initiated from casting pores (Douglas, 1999; Dennis, 2000; MacLachlan and Knowles, 2001a). Fig. 2(a) shows a fracture surface from a nickel superalloy tested under creep conditions (950 °C) with facet cracks and likely initiation sites. A typical crack, which appears to emanate from a pre-existing casting pore, is shown in Fig. 2(b); Fig. 2(c) illustrates the proposed mechanism of crack propagation from a relatively blunt notch. Due to localised deformation in the vicinity of the notch root, microcracks are expected to initiate, either at the notch, or at a neighbouring pore. Under increasing load, these microcracks propagate and coalesce, leading to the growth of the mesoscopic crack and eventual fracture of the specimen. Further evidence of the initiation of micro-cracks at casting pores in Ni-base single crystal superalloys has been obtained in the low cycle fatigue tests on RR2000 and CMSX4 at 750 °C and on directionally solidified Ni-base superalloys at 650 °C by MacLachlan and Knowles (2001b) and Okazaki et al. (1990), respectively.

An additional source of heterogeneity at the microstructural length scale may arise from the material degradation caused by local oxidation, which becomes significant for uncoated Ni superalloys at temperatures in excess of ~800 °C (Gobel et al., 1993; Bouhanek et al., 1997; Gross et al., 1998; Li et al., 2003). Generally, oxidation leads to the degradation of materials in two ways. First, a brittle oxide layer, which is more vulnerable to cracking, forms on the material surface. Cracks initiating in the oxide may propagate into the substrate and lead to failure (Reuchet and Remy, 1983; Nue and Sehitoglu, 1989;
MacLachlan and Knowles, 2001b; Ohtani et al., 2001). Second, due to oxidation-linked diffusion and interdiffusion processes in the bulk alloy, the phase composition of the material is altered, which introduces a microstructural change in the bulk material beneath the oxide (Nesbitt, 1982; Lerch and Antolovich, 1990; Ohtani et al., 2001). The local degradation in the material microstructure modifies the material constitutive behaviour which in turn can lead to a reduction in the time to crack initiation (Dumoulin et al., 2003). The effects of oxidation on fatigue life have been investigated experimentally in Ni-base superalloys under thermo-mechanical loading. MacLachlan and Knowles (2001b) showed that during low cycle fatigue of the commercial superalloys RR2000 and CMSX4 at 850 and 950 °C, fatigue cracks may initiate from oxide spikes on the surface. This phenomenon was also observed in tests by Ohtani et al. (2001) on the nickel-base single crystal superalloy CMSX10 at 1000 °C. Previous numerical studies on oxidation effects in nickel alloys and thermal barrier coatings have concentrated on the effect of the property mismatch between the oxide scale and the substrate. Failure models, based on crack propagation driven by internal stresses due to property mismatch and oxidation effects, were proposed to predict the lifetime of thermal barrier coating systems (Chang and Phucharoen, 1987; Busso et al., 2001a; Sfar et al., 2002; Tomimatsu et al., 2003). However, these analyses did not consider the change in composition of the system due to diffusion and interdiffusion, linked to the oxidation process. Recently, a coupled diffusion–deformation approach was proposed in Dumoulin et al. (2003) to study the effect of oxidation on the deformation and fracture behaviour of a commercial nickel superalloy. The approach relied on a crystallographic model to describe the macroscopic (homogenised) constitutive behaviour of the two-phase single crystal and a mass diffusion model to describe the diffusion and interdiffusion processes associated with oxidation of the alloy.

Fig. 2. (a) Micrograph of square facets believed to have originated at a casting pore (indicated by arrows) from a [010] creep test at 950 °C (Dennis, 2000), (b) a typical cracked pore (Dennis, 2000) and (c) schematic of crack propagation from a blunt notch by microvoid coalescence.
A damage model, based on the initiation of microcracks from the surface of internal voids at the mesoscale (~20 μm) was incorporated within this framework. It was found that, due to the degradation of the material behaviour local to the notch, the time to initiate a crack in a notched compact tension specimen in an oxidising environment may be a factor of three lower than that in vacuum. However, in that work, the interaction between the void and crack was not accounted for as the void was not modelled explicitly. In the current work, the deformation fields for a microvoid in the vicinity of a relatively sharp notch in a single crystal nickel alloy are examined. An idealised cylindrical microvoid, representing a material casting defect in a notched compact tension specimen, is considered and a strain based failure criterion is used to predict the crack initiation time under creep loading. The paper is laid out as follows: Sections 2 and 3 describe the mass diffusion and crystallographic models used in the analysis. Sections 4 and 5 outline the finite element procedures and the crack initiation criteria, respectively. The results of the analysis are described in Section 6, including the effect of environment, load level and void location on the time to crack initiation. In Section 7, a generic, probabilistic approach to the prediction of crack initiation in nickel-base superalloys is proposed. Finally, concluding remarks are presented in Section 8.

2. Mass diffusion model

2.1. Oxidation of two-phase Ni–Cr–Al alloys

Oxidation phenomena in two phase (γ + γ') Ni–Cr–Al alloys have been discussed in detail in Dumoulin et al. (2003). The commercial superalloy CMSX4 has a relatively low Cr content (7.5 at.%) and a high Al content (19.7 at.%). Thus, following the early stages of oxidation, a continuous and stable Al2O3 layer forms on the alloy surface (Bouhanek et al., 1997; Gross et al., 1998). As the aluminium is expended to form the oxide, a non-equilibrium state exists in the (γ + γ') alloy leading to the dissolution of the Al-rich γ' precipitates. As oxidation progresses, the γ' precipitates continue to dissolve and two distinct regions develop: a pure γ region near the surface and an unaffected (γ + γ') region within the bulk of the crystal. A micrograph illustrating the depleted γ region surrounding

![Fig. 3. Oxide-filled crack and depleted γ region surrounding a crack in RENE N4 following exposure to an oxidising environment for approximately 10 h at 1093 °C (adapted from Lerch and Antolovich, 1990).](image-url)
a crack in a Ni-base superalloy RENE N4 at 1093 °C is shown in Fig. 3 (from Lerch and Antolovich, 1990). It may be noted that the size of the γ region is on the order of 5 μm. The time at which this micrograph has been taken is not stated in the paper but we have inferred it to be approximately 10 h (based on a life of 20,000 cycles with a frequency of 0.5 Hz).

Experimental measurements reveal that the growth rate of the surface oxide formed on CMSX4 follows a parabolic law (Bouhanek et al., 1997). Therefore, the flux of aluminium from the metal into the oxide, $J_{Al}$, at time, $t$, can be expressed as (Nesbitt, 1982):

$$J_{Al} = -\lambda(1 - V_{Al}C_{Al}^{i})\sqrt{\frac{\kappa_p}{t}}, \quad (1)$$

where $\kappa_p$ is the parabolic rate constant for oxidation, $C_{Al}^{i}$ the Al concentration at the alloy-oxide interface, $V_{Al}$ the partial molar volume of Al and $\lambda$ a material-dependent parameter. For CMSX4, $\lambda = 0.125 \text{mm}^3/\text{mg}$ and $1/V_{Al} = 81.8 \text{at.}\%$ (see Dumoulin et al., 2003).

In conjunction with the diffusion of Al, Ni diffuses towards the γ region due to the dissolution of the precipitates while Cr diffuses away from the oxide/γ interface and tends to accumulate near the γ/(γ + γ′) interface. The diffusion of Ni and Cr are not modelled here as these elements play a secondary role in the formation of the oxide and the dissolution of the γ′ precipitates. Furthermore, the oxide scale will not be explicitly modelled in this study as the oxide thickness is expected to be small compared to the other important length scales of the problem (such as the void spacing or thickness of the γ region). The effect of the oxide scale on crack initiation in single crystal alloys will be presented in a separate publication (Kubler et al., 2006).

2.2. Finite element implementation of the diffusion problem

Numerical modelling of atomic diffusion in systems containing a moving material interface requires the solution of Fick’s first and second laws, constrained by the mass balance at the moving interface and subject to the flux boundary condition given by Eq. (1). In the finite element mass diffusion analysis carried out in this work, the position of the moving γ/(γ + γ′) interface boundary is not determined explicitly. Instead, the interface boundary is introduced implicitly by selecting the value of the diffusion coefficient, $D_{Al}$, on the basis of whether the material point is within the γ region or the (γ + γ′) region. If the concentration of aluminium is below the upper solubility limit of Al in γ (12.5 at.% for CMSX4 at 950 °C), then the material point is designated to be in the γ region and the appropriate diffusion coefficient is assigned. Otherwise, $D_{Al}$ is taken to be zero and it is assumed that no diffusion takes place within this region (it has been seen (see e.g. Nesbitt, 1982) that the diffusivity of aluminium in the (γ + γ′) region is much less than that in the γ region). In this way, the position of the interface is determined from the analysis as the location where the upper solubility limit is reached. This approach, which requires the specification of an initial, negligibly small γ layer at the start of the analysis, has been validated by the close agreement between a 1D FE solution of a moving interface problem with the corresponding finite difference solution taking full account of the motion of the interface (see Dumoulin et al., 2003, for details).
3. Crystallographic single crystal material model

The macroscopic mechanical constitutive behaviour of the superalloy of interest, CMSX4, is described by a finite deformation, multiscale rate dependent crystallographic formulation (Dennis, 2000) which extends the physically-based model of Busso (1990) and Busso and McClintock (1996). The formulation relies on the multiplicative decomposition of the total deformation gradient, \( \mathbf{F} = \mathbf{F}^e \mathbf{F}^p \), where \( \mathbf{F}^e \) is the component which accounts for elastic deformation and rigid-body rotations and \( \mathbf{F}^p \) is the inelastic component associated with pure slip. \( \mathbf{F}^p \) is calculated from the plastic velocity gradient, \( \mathbf{L}^p \), given as,

\[
\mathbf{L}^p = \dot{\mathbf{F}}^p \dot{\mathbf{F}}^p^{-1} = \sum_{\alpha=1}^{n} \dot{\gamma}^\alpha (\mathbf{m}^\alpha \otimes \mathbf{n}^\alpha),
\]

where \( \dot{\gamma}^\alpha \) is the shear strain rate on the slip system \( \alpha \) and \( \mathbf{m}^\alpha \) and \( \mathbf{n}^\alpha \) are the slip direction and the slip plane normal, respectively (see e.g. Asaro, 1983).

Based on a stress-dependent activation energy, the flow rule is expressed in terms of two scalar state variables per slip system \( \alpha \): a macroscopically average slip resistance, \( S^\alpha \), and an internal or back stress, \( B^\alpha \). Thus,

\[
\dot{\gamma}^\alpha = \dot{\gamma}_0 \exp \left[ -\frac{F_0}{k\theta} \left( 1 - \left| \frac{\tau^\alpha - B^\alpha}{\tau_0^\alpha \mu / \mu_0} \right|^p \right)^q \right] \text{sgn}(\tau^\alpha - B^\alpha),
\]

where \( k \) is the Boltzmann constant, \( \tau^\alpha \) is the resolved shear stress on the system \( \alpha \), \( \theta \) the absolute temperature, \( \mu, \mu_0 \) the shear moduli at \( \theta \) and 0 Kelvin, respectively, and \( F_0, \tau_0, p, q, \dot{\gamma}_0 \) are material constants.

In this work, the slip resistance in each slip system, \( S^\alpha \), is related to the overall dislocation density for that system, \( \rho^\alpha \), by the relationship,

\[
S^\alpha = \lambda \mu b^\alpha \left( \sum_{\beta=1}^{N} \delta_{S}^{\alpha\beta} \rho^{\beta} \right)^{1/2},
\]

where \( \lambda \) is a parameter which accounts for the nature of the dislocation distributions, \( b^\alpha \) is the magnitude of the Burgers vector, \( \delta_{S}^{\alpha\beta} \) the cross hardening or interaction matrix and \( N \) is the number of slip systems.

The evolutionary behaviour of the overall dislocation density and the associated back stress for the slip system \( \alpha \) are expressed as,

\[
\dot{\rho}^\alpha = \frac{1}{b^\alpha} \left( \frac{1}{l_{app}^\alpha} - d_D \rho^{\beta} \right) |\dot{\gamma}^\alpha|,
\]

\[
\dot{B}^\alpha = h_B |\dot{\gamma}^\alpha| - r_D B^\alpha |\dot{\gamma}^\alpha|.
\]

In Eq. (5) \( l_{app}^\alpha \) is the dislocation mean free path, (an average measure of the distance travelled by a dislocation between source and sink (storage) points) and \( d_D \) is a dynamic recovery coefficient. In Eq. (6), \( h_B \) is a hardening coefficient and \( r_D \) is a dynamic recovery function, which depends on the current state given by \( \rho^\alpha \). The multiscale nature of the formulation is incorporated into Eqs. (5) and (6) by making an explicit link between the initial dislocation density, \( \rho_0^\alpha \), and the dislocation mean free path, \( l_{app}^\alpha \), with the features of the microstructure at a lower scale, i.e. at the micro-scale. Thus, for the current material
system, the functional forms for $\rho_0^z$ and $l^z_{app}$ are defined in terms of the size, $D$, and volume fraction, $v_f$, of the $\gamma'$ precipitate population,

$$\rho_0^z = \hat{\rho}_0^z(D/D_m, v_f, \theta),$$

$$l^z_{app} = l^z_{S}(D/D_m, v_f, \theta),$$

(7)

where $D_m$ is a reference particle size (here taken as the mean CMSX4 precipitate size, $D_m = 0.5\, \mu m$) and $l^z$ is the mean dislocation spacing, given as,

$$l^z = \left(\sum_{\beta=1}^{N} \delta^z_{\beta} \rho^\beta \right)^{-1/2}.$$  

(8)

The calibration of the functional forms in Eq. (7) has been based on experimental data in conjunction with FE studies at the microscale, which incorporated the relevant details of the material microstructure (Busso et al., 2000; Meissonnier et al., 2001). Thus, through Eqs. (3)–(8), the crystallographic model can account for the changes in the macroscopic stress–strain response resulting from changes in precipitate size and volume fraction linked to local kinetic processes (i.e. interdiffusion, oxidation).

The crystallographic formulation has been implemented numerically as a user defined material subroutine within the commercial finite element code ABAQUS (2003). The numerical implementation relies on an implicit algorithm to update the stress and the internal slip system variables (see Busso et al., 2000; Meissonnier et al., 2001). The calibration of the material model was carried out under the assumption that twelve octahedral ($\{1 1 1\}^{h}011_i$) and six cubic ($\{1 0 0\}^{g}011_i$) slip systems are potentially active. The reference data used to calibrate the model for the initial precipitate volume fraction, $v_f = 68\%$, were obtained from uniaxial monotonic, cyclic and creep tests and those for $v_f < 68\%$ were generated from 3D finite element analyses of periodic unit cells of representative volume elements (Busso et al., 2000). The model has been shown to provide a good prediction of the material thermo-mechanical behaviour over a wide range of loading rates and temperatures (Dennis, 2000).

As discussed in Section 2.1, the diffusion and interdiffusion processes linked to surface oxidation result in the local dissolution of the $\gamma'$ precipitates, leaving a pure $\gamma$ phase, that is, $v_f = 0\%$. The effect of $\gamma'$ precipitate volume fraction, $v_f$, on the uniaxial monotonic and steady-state cyclic response in the $\langle 0 0 1 \rangle$ direction at 950°C is shown in Figs. 4(a) and (b), respectively. Thus, the crystallographic model presented here can account for the change in mechanical response resulting from changes in volume fraction linked to oxidation and diffusion processes at high temperatures.

4. Finite element procedure

A typical 2D finite element (FE) mesh for the compact tension specimen is shown in Fig. 5. The specimen contains a semi-circular notch with radius, $R = 0.1\, \text{mm}$, and the notch length to specimen width ratio, $a/W$, is 0.5. As indicated in Fig. 5(a), the plane of the specimen is normal to the [0 0 1] crystallographic axis and the crack lies on the (0 1 0) plane. Due to the symmetries of the specimen and the crystallographic lattice, only half of the specimen is modelled.
The void density of the alloy is low with typical pore spacing on the order of 100 µm (see Fig. 2a). Therefore, in this work, void interaction is ignored and only the interaction of a single void with the notch is examined. The void, indicated in Fig. 5(b), has a diameter $d = 15 \mu m$ and is located a distance $L/d = 10$ from the notch.

![Fig. 4. Effect of precipitate volume fraction, $v_f$, on (a) the \textit{h}001\textit{i} uniaxial monotonic response and (b) the \textit{h}001\textit{i} steady-state uniaxial cyclic response at 950°C and at constant strain rate (the experimental data, indicated by the symbols, are from Dennis, 2000).](image4)

![Fig. 5. (a) Finite element mesh for the CT specimen; (b) refined mesh around the notch and the void; (all dimensions in mm).](image5)

The void density of the alloy is low with typical pore spacing on the order of 100 µm (see Fig. 2a). Therefore, in this work, void interaction is ignored and only the interaction of a single void with the notch is examined. The void, indicated in Fig. 5(b), has a diameter $d = 15 \mu m$ and is located a distance $L/d = 10$ from the notch.
A fine mesh is generated around the notch and the void as shown in Fig. 5(b). The whole mesh consists of \( \approx 3500 \) four-node elements and the smallest element size near the void is approximately \( 1.5 \mu m \), close to the size of \( \gamma' \) precipitates in CMSX4 (mean size typically \( 0.5 \mu m \)). Mesh sensitivity studies have been carried out and it has been found that a four-fold increase in the mesh density close to the notch and void leads to a maximum difference in the predicted crack initiation time for the original and refined mesh of approximately 10%. In view of the increased computation times for the refined mesh and the relatively small improvement in accuracy, the majority of the results presented here were obtained using the mesh shown in Fig. 5.

To solve the overall oxidation–deformation problem, a fully coupled mass–diffusion–mechanical analysis was performed. The aluminium concentration profile determined from the mass diffusion analysis is used to assign the material behaviour at each integration point. If the concentration is above the upper solubility limit of aluminium in \( \gamma \), the material properties are those of \( (\gamma + \gamma') \); otherwise they are those of pure \( \gamma \). The advantage of a fully coupled analysis (as opposed to the sequentially coupled analysis carried out by Dumoulin et al., 2003) is that the effect of geometry change (e.g. notch opening) on the diffusion can be taken into account. A coupled analysis also has the potential to include the dependence of diffusivity on the stress state. However, the majority of work investigating the effect of hydrostatic stress on diffusion has focused on the diffusion of hydrogen in structural steels (e.g. Krom et al., 1999) and there is relatively little information available for diffusion of oxygen and aluminium in nickel alloys. Therefore, this effect has not been included in the current work, which may lead to an underestimate of the rate of growth of the pure \( \gamma \) region and an overestimate of the failure times, when oxidations effects are important.

The analysis is conducted at a constant temperature of \( 950^\circ C \). The load, \( P \), is applied to the specimen through a rigid pin with the pin and specimen modelled as a pair of contact surfaces. Analyses have been conducted for values of load, \( P = 4, 5.5, 7, 8.5 \) and \( 10 \) kN, corresponding to stress intensity factors for the notched specimen of \( K = 20, 27, 35, 42 \) and \( 50 \) MPa√m, respectively.

5. Crack initiation criteria

A void growth model was formulated by Dennis (2000) and Busso et al. (2001b) for CMSX4 under creep conditions, using the multiscale rate-dependent crystallographic model presented here. A fracture initiation criterion was also proposed in Dennis (2000), based on the assumption that a microcrack will initiate in the vicinity of a void or notch when a critical value of the equivalent inelastic strain is reached at the void surface, i.e.,

\[
\tilde{\varepsilon}_p = \tilde{\varepsilon}_p^{\text{critical}},
\]

where \( \tilde{\varepsilon}_p \) is the equivalent inelastic strain and \( \tilde{\varepsilon}_p^{\text{critical}} \) is a material property. The equivalent strain measure, \( \tilde{\varepsilon}_p \), is calculated from:

\[
\tilde{\varepsilon}_p = \sqrt{\frac{2}{3}(F_p - I)(F_p - I)},
\]

where \( F_p \) is obtained from Eq. (2) and \( I \) is the identity matrix.

In Dumoulin et al. (2003), a void growth formulation based on the results of Dennis (2000) and a crack initiation criterion were implemented numerically as a damage variable.
to predict microcrack initiation in an oxidising CT notch specimen under creep loading conditions. The effect of the void was incorporated through the damage variable and thus the interaction between the void and the notch, which may be important, particularly at small void distances, was not accounted for. Here, the void is modelled explicitly and the void-notch interaction and the effect of void distance from the notch on the deformation are examined directly.

In this work, crack initiation is deemed to occur when the equivalent inelastic strain $\varepsilon^p$ reaches the critical value $\varepsilon^p_{\text{critical}}$ over a characteristic distance, $d^*$. The use of a critical strain in conjunction with a critical distance has been widely used as a ductile fracture criterion, e.g. Riedel and Rice (1980); Ritchie and Thompson (1985). The choice of characteristic distance introduces a physical length scale, associated with the fracture process, and avoids issues associated with the stress and strain singularity ahead of a sharp crack tip. In the current work, although a rather blunt notch is studied (notch radius to crack length, $R/a = 8 \times 10^{-3}$), the strain gradient in the vicinity of the notch and void are relatively large. Therefore, there is a need to specify a characteristic distance when applying the failure criterion. The chosen distance $d^*$ is 1.5 $\mu$m which is on the order of the spacing of the $\gamma'$ precipitates. Hence failure at the microscale can be associated with micro-crack growth and coalescence within a number of $\gamma$ channels (see Fig. 6). The distance 1.5 $\mu$m also corresponds to the size of one element adjacent to the void in the FE mesh, as discussed in Section 4. It will be seen in Section 6.2 that the results are not very sensitive to the value of $d^*$ at the meso-scale (scale of the void). The value of the local critical failure strain, $\varepsilon^p_{\text{critical}}$, for CMSX4 was found to be 0.38 through a combination of FE analyses and experimental measurements of a notched bar specimens under creep loading (Dennis, 2000). For a given temperature, this value is assumed to be a material property, independent of, for example, the local strain rate. Note also that in this work the critical failure strain $\varepsilon^p_{\text{critical}}$ is not a function of stress triaxiality. Stress triaxiality effects on (macroscopic) failure strain are generally accounted for through the use of analytical models which account for growth and coalescence of microvoids, e.g. Ritchie and Thompson (1985), Budden and Ainsworth (1999). In this work the effect of the void on local deformation and fracture is included explicitly and the failure criterion is applied at

Fig. 6. Micro-crack growth in the vicinity of a pore (adapted from Douglas, 1999). It may be noted that crack growth occurs primarily within the $\gamma$ channels.
the mesoscale (i.e. scale of the void). Thus, adjustment to the failure strain to account for stress triaxiality is not needed.

With the proposed criterion, the time to crack initiation for a notched CT as examined for air and vacuum conditions under constant load (creep loading).

6. Results of the computational analyses

6.1. Diffusion analysis

The results of a typical FE diffusion simulation of the CT specimen is illustrated in Fig. 7. The dark area of the specimen shown in Fig. 7(a) is the predicted γ region after 100 h exposure to oxidation. At this time, the region has a thickness of ~25 μm. In Fig. 7(b) the size of the γ region as a function of the oxidation rate constant $k_p$ is provided for three different times. It may be seen that the thickness of the γ region depends strongly on $k_p$, which controls the aluminium flux rate from the alloy into the oxide (see Eq. (1)). The value of $k_p$ for CMSX4 at 950°C in air was taken to be $2.84 \times 10^{-11}$ mg²/mm⁴/s (based on the measured oxide growth profile reported in Rieck, 1999). Due to this relatively low value of $k_p$, the thickness of the γ region for CMSX4 is quite limited for oxidation times shorter than 1000 h, as seen in Fig. 7(b). Also shown in Fig. 7(b) is the predicted size of the γ region for another single crystal nickel superalloy (SC16), which has a higher oxidation rate constant ($k_p = 2.4 \times 10^{-10}$ mg²/mm⁴/s) and a correspondingly thicker γ region. Note that the value of $k_p = 8.0 \times 10^{-10}$ mg²/mm⁴/s used in the analysis of Dumoulin et al. (2003) is relevant to the Ni–Cr–Al alloys with higher Al contents studied by Nesbitt (1982). Those analyses are expected to overpredict the rate of oxidation and the size of the γ region for CMSX4.

Fig. 7. Oxidation near the notch: (a) plot of the predicted γ' depleted region after 100 h oxidation for $D_{Al} = 3.47$ mm²/s, $k_p = 8.0 \times 10^{-10}$ mg²/mm⁴/s, (b) the effect of the oxidation rate constant $k_p$ on the growth of the γ region, (the predicted results for two Ni superalloys (CMSX4 and SC16) are indicated on the figure).
6.2. Crack initiation analysis

Results are presented for the case of constant load (creep) conditions. In future work, the approach will be applied to cyclic load (low cycle fatigue) conditions, Zhao et al. (2006). The crack initiation time has been determined using the criterion given by Eq. (9) with and without oxidation and for different load levels, quantified by the stress intensity factor, $K$, and varying distance of the void from the notch, $L/d$.

6.2.1. Crack initiation under constant load—vacuum conditions

Results are first presented for the case when oxidation does not occur (vacuum conditions). Fig. 8 shows contours of equivalent inelastic strain after 400 h for a CT specimen with $K = 35 \text{ MPa} \sqrt{m}$ and a representative void distance of $L/d = 10$. In the analysis, the load was linearly increased to the maximum value in 100 s and maintained at this level for the duration of the analysis. It is seen in Fig. 8 that significant inelastic strain accumulates at the void surface, indicating that crack initiation is expected to occur near the void.

The predicted crack initiation time for CMSX4, with $\varepsilon^p_{\text{critical}} = 0.38$, is plotted in Fig. 9, as a function of the applied loading level. The critical strain is reached at a location adjacent to the void and one or two elements away from the specimen symmetry line, which indicates that the microcracks tend to initiate in a direction approximately normal to the applied load as observed in experiments (Douglas, 1999; Dennis, 2000; MacLachlan and Knowles, 2001a). It may be seen that the predicted life reduces significantly with increasing load. It is found that if $K > 40 \text{ MPa} \sqrt{m}$, fracture initiation occurs during loading, that is when $t < 100$ s.

The effect of the void spacing, $L/d$, is also shown in Fig. 9. For $L/d = 2$, the void is very close to the notch and the time to crack initiation is significantly reduced. This is due to the
strong interaction between the notch and the void as seen in Fig. 10—a concentrated shear band emanates from the notch and amplifies the strain at the void. At $K = 20 \text{ MPa} \sqrt{m}$, an approximately 10-times reduction in the time to crack initiation is predicted when the void location is changed from $L/d = 10$ to $L/d = 2$. For $K = 35 \text{ MPa} \sqrt{m}$, the critical strain is reached at the void surface soon after the application of the load—the predicted initiation time is 103 s and for $K > 35 \text{ MPa} \sqrt{m}$, a microcrack is predicted to initiate almost immediately after the load is applied.
The predictions from the damage-based analysis of Dumoulin et al. (2003) are also included in Fig. 9. In the work of Dumoulin et al., the time to crack initiation was taken as the time when the fully damaged region extended 50 \( \mu \text{m} \) ahead of the notch. A damage region of 50 \( \mu \text{m} \) in that work can be considered to be equivalent to a void spacing of \( L = 50 \mu \text{m} \) for the current analysis. It can be seen in Fig. 9 that the results from Dumoulin et al. (2003) (dash line) are located between those for \( L/d = 2 \) (\( L = 30 \mu \text{m} \)) and \( L/d = 10 \) (\( L = 150 \mu \text{m} \)) and are therefore consistent with the current analysis.

The above results are based on the condition that microcracks start to initiate when the critical inelastic strain is reached over a distance of \( d^* = 1.5 \mu \text{m} \) from the void surface as discussed in Section 5. Fig. 11 examines the effect of the choice of critical distance on the time to crack initiation. The results for two values of critical distance, \( d^*/d \), are examined, \( d^*/d = 0.1 \) and 0.05 which correspond to 1.5 and 0.75 \( \mu \text{m} \), respectively. The refined mesh discussed in Section 4 has been used for these analyses since the element size must be less than or equal to \( d^* \). It may be seen that although a shorter initiation time is predicted when a smaller distance \( d^* \) is considered, the result is not strongly sensitive to \( d^* \) at these length scales.

6.2.2. Comparison with the Riedel–Rice K-based creep failure assessment

The contour plots of accumulated inelastic strain in Figs. 8 and 10 show that the creep deformation is highly localised in a small zone around the notch and void before crack initiation occurs. Relative to the specimen dimensions, the creep zone is very limited in size and thus the problem falls into the small scale creep regime. Under small-scale creep conditions, Riedel and Rice (1980) proposed a relation to predict the initiation of a stationary crack in an isotropic, power law creep material, based on the linear elastic stress intensity factor \( K \). It was assumed that initiation occurs when the critical equivalent inelastic strain, \( \varepsilon^c \), is reached at a small distance, \( r \), ahead of the crack tip. The time to

![Figure 11. Comparison of crack initiation times for two critical distances, \( d^* \).](image-url)
crack initiation, \( t_i \), is then given by

\[
t_i = \frac{(\dot{\varepsilon}^c)^{n+1}}{E^n B(n+1) K^2 (1 - \nu^2)} \left[ \frac{E^2 I_n r}{K^2 (1 - \nu^2)} \right]^n,
\]

where \( E \) is the (isotropic) Young’s modulus, \( \nu \) is the Poisson’s ratio, \( B \) and \( n \) are parameters of the isotropic power-law creep relation, \( \dot{\varepsilon} = B \sigma^n \), measured in uniaxial tension creep tests. The value of \( I_n \) depends on the creep exponent \( n \) (see e.g. Shih, 1974). Eq. (11) has been derived for an isotropic material with a simple power law creep dependence. The applicability of the approach to the more complex material response of a single crystal alloy is examined here.

For CMSX4, the steady-state creep deformation in the \( <001> \) direction at 950 °C is shown in Fig. 12(a), where the high and low stress regimes are considered separately. The creep parameter values were obtained from a least-square curve fitting of the test data, with \( B = 1.88 \times 10^{-20} \text{MPa}^{-n} \text{s}^{-1} \) and \( n = 4.72 \) in the low stress regime and \( B = 2.12 \times 10^{-56} \text{MPa}^{-n} \text{s}^{-1} \) and \( n = 17.93 \) in the high stress regime. The corresponding values of \( I_n \) are 5.1 and 4.2 for the low and high stress regimes, respectively (Shih, 1974).

Eq. (11) was used to determine the crack initiation time for the CT specimen. The Young’s modulus, \( E \), and the Poisson’s ratio, \( \nu \) are the \( <001> \) values for CMSX4 at 950 °C and \( r \) is the distance of the void from the notch root. The predicted crack initiation times from the Riedel and Rice (RR) model are compared with the result from the FE analyses in Fig. 12(b), where the assumed critical strain values, \( \varepsilon^c \), are \( \sim 0.05 \) for \( L/d = 10 \) and \( \sim 0.1 \) for \( L/d = 2 \). Note that the failure strain in Eq. (11), which is determined here by fitting to the FE results, is a macroscale quantity and thus is not expected to be equal to the mesoscale failure strain, \( \varepsilon^{p, \text{critical}} \), which includes the effect of the void at the meso/micro scale.

A good correspondence between the Riedel–Rice relation and the FE result is demonstrated in Fig. 12(b). Thus, it is seen that when small scale creep conditions prevail, the time to crack initiation in the single crystal material may be characterised using the linear elastic stress intensity factor, in conjunction with an (isotropic) power law representation of the material response. It is also interesting to note that when plotted in

![Fig. 12. (a) Steady-state creep deformation in \( <001> \) direction for CMSX4 at 950 °C and (b) comparison between Riedel–Rice equation (Eq. (11)) and FE analysis for crack initiation time under creep loading conditions.](image-url)
the form of Fig. 12(b), a distinct low stress and high stress regime can be identified in the FE predictions of the crack initiation time. Thus, it is seen that reasonable predictions are obtained using the Riedel–Rice relation if the anisotropic response of the single crystal alloy is replaced by an equivalent, isotropic power law response in the appropriate stress regime. Note that the good agreement between the FE prediction and Eq. (11) was obtained by using different critical strains corresponding to the different void locations. The requirement for different macroscale failure strain values to match the FE predictions for different void locations may be related to changes in stress triaxiality at the different locations. This issue will be examined further in future work.

6.2.3. Crack initiation under constant load—effects of oxidation

As discussed in Section 6.1, oxidation of CMSX4 leads to the formation of a (soft) γ region adjacent to the notch root. The local reduction in strength of this region will result in the redistribution of stress (and strain) in the vicinity of the notch. These effects are

![Graphs showing stress distribution under oxidation](image)

Fig. 13. (a) Distribution of the [010] stress component along the specimen symmetry line for \( L/d = 10 \) at \( K = 35 \text{ MPa}\sqrt{m} \) at different times, (b) distribution of [010] stress when effects of oxidation are accounted for, (c) comparison of [0 1 0] stress with and without oxidation after 400 h, (d) comparison of equivalent inelastic strain, \( \varepsilon_p^e \), with and without oxidation after 400 h. Here \( \kappa_p = 8.0 \times 10^{-10} \text{ mg}^2/\text{mm}^4/\text{s} \).
illustrated in Figs. 13(a) and (b), which show the evolution of the stress distribution in the notch vicinity for \( L/d = 10 \) with and without oxidation.

In Fig. 13 stress and strain are plotted as a function of distance, \( r \), from the notch root; a distance \( r/L = 1 \) corresponds to the void surface (see inset to Fig. 13a). Note that the peak stress is initially located close to the notch root (\( r/L = 0 \)) but following the redistribution of stress due to creep (in Fig. 13a) and creep and oxidation (in Fig. 13b), the peak stress shifts towards the void, (\( r/L = 1 \)). A direct comparison between the stresses with and without oxidation is provided in Fig. 13(c). It may be seen that due to oxidation and the formation of the low strength \( \gamma \) layer, the stress is significantly reduced in the vicinity of the notch (from 400 to 50 MPa at the notch root after 400 h) and slightly increased near the void (from 460 to 500 MPa after 400 h). Fig. 13(d) shows that, in addition to this reduction in stress, there is an increase in accumulated inelastic strain near the notch and the void. The peak inelastic strain is still located in the vicinity of the void (increasing from 0.39 to 0.47 at \( r/L = 1 \)) and crack initiation is again predicted to occur at the void (note that this does not take into account any possible change in material ductility, \( \varepsilon/\varepsilon_{\text{critical}} \), due to the formation of the \( \gamma \) layer).

The effect of oxidation on the predicted time to crack initiation is shown in Fig. 14. The magnitude of the reduction in life due to oxidation depends on the oxide growth rate constant \( \kappa_p \) and the loading level, quantified by the stress intensity factor, \( K \), for a given void location. Lower values of \( K \) and higher values of \( \kappa_p \) lead to a greater reduction in the predicted life time. Fig. 14(a) shows the predicted time to microcrack initiation as a function of \( K \), for \( L/d = 10 \) with \( \kappa_p = 2.84 \times 10^{-11} \text{mg}^2/\text{mm}^4/\text{s} \) (the value for CMSX4). As expected, the time to crack initiation is reduced, if the effects of oxidation are included. Fig. 14(a) also shows that the effect is reduced with an increase in load, since the life-time is significantly shortened, which in turn reduces the time for oxidation to occur. The sensitivity of the crack initiation time to \( \kappa_p \), the oxide growth rate constant, is shown in Fig. 14(b) for \( L/d = 10 \) and \( K = 35 \text{MPa}\sqrt{\text{m}} \). An increase in \( \kappa_p \) increases the oxidation and diffusion rates, leading to earlier crack initiation. For CMSX4, the time to crack initiation at this load is only slightly reduced due to the relatively low value of \( \kappa_p \).
7. Discussion and interpretation of results

The FE studies have shown that the crack initiation time for the nickel superalloy depends strongly on the applied loading level and the distance of the void from the notch. It has also been found that the results can be described with the Riedel–Rice (RR) small scale creep crack initiation model and the linear elastic stress intensity factor, $K$.

The applicability of the RR model provides the potential for a probabilistic-based framework for the prediction of initiation time for CMSX4, which incorporates the strong sensitivity to void location. If the distance of the void from the notch, $L$, is represented as a probability distribution function, i.e. the probability that $L$ is less than a certain value, then as shown below the probability distribution function for the time to failure can be obtained. The Riedel–Rice relation, Eq. (11), can be rewritten as

$$t_i = \frac{(\frac{\varepsilon}{C_1})^{n+1}}{E^2 B(n+1)} \left[ \frac{E^2 I_n}{K^2 (1 - v^2)} \right]^n = \left( \frac{A_n}{K^n} \right)^n,$$

where, for simplicity, we take the failure strain $\varepsilon$ to be independent of $r$ and, for a fixed temperature,

$$A_n = \frac{(\frac{\varepsilon}{C_1})^{n+1}}{E^2 B(n+1)} \left[ \frac{E^2 I_n}{(1 - v^2)} \right]^n$$

depends only on the creep exponent, $n$.

Writing the probability density function (pdf) for $r$ as $f(r)$, then following standard rules for pdfs, the pdf of initiation times, $g(t_i)$, from Eq. (12) is,

$$g(t_i) = f\{K^2(t_i/A_n)^{1/n}\} \frac{dr}{dt_i}.$$

Depending on the form of $f(r)$ it may be possibly to obtain the pdf $g(t_i)$ and associated probability distribution function in closed form. For example, if the distance, $r$, in the RR model is taken to be the distance from the notch to the nearest void, $L$, (as in Section 6.2.2) and if this distance is described by an exponential law,

$$f(r) = \lambda e^{-\lambda r}$$

which has mean and standard deviation $1/\lambda$, then the pdf of initiation times, $g(t_i)$, is given by,

$$g(t_i) = \frac{\lambda K^2}{nA_n^{1/n}} e^{-\lambda K^2(t_i/A_n)^{1/n}} t_i^{(1-n)/n}.$$

To find the probability distribution function of the failure time, $P(t_i \leq T)$, the pdf, $g(t_i)$, is integrated to give

$$P(t_i \leq T) = \int_0^T g(t) \, dt = 1 - e^{-(\lambda K^2/A_n)^{1/n} T^{1/n}}.$$

For more complex forms of the pdf, $f(r)$, the differentiation and integration in Eqs. (14) and 17 can be carried out numerically.

The probability distribution function for the initiation time (Eq. 17) clearly depends on the creep exponent, $n$. As an example we first consider the case, $n = 4.72$ (which corresponds to the low stress regime in Fig. 12a). Fig. 15 illustrates the probability...
distribution function for the void spacing, \( r \), using an exponential density function with 
\( 1/\lambda = 90\mu m \). It may be seen that for this value of \( \lambda \) the median value, \( P(r \leq R) = 0.5 \) is 63\( \mu m \) and that the probability that \( r \) lies between 15 and 150\( \mu m \) (i.e. \( 1 \leq L/D \leq 10 \)) is approximately 66\%. The corresponding distribution of initiation times is illustrated in Fig. 16(a) for two load levels, \( K = 20 \) and \( K = 30 \) MPa\( \sqrt{\mu m} \). The macroscopic failure
strain, $\varepsilon^c$, has been taken to be 0.075 which is the average of the two failure strains used in Fig. 12(b). It may be seen that the analysis predicts that the median initiation times are $6 \times 10^3$ h and 150 h for $K = 20$ and 30 MPa $\sqrt{m}$, respectively, which is consistent with the trends shown in Fig. 12(b).

The FE result shown in Fig. 12(b) suggests that for large values of $K$ ($K \geq 28$ MPa $\sqrt{m}$) and small values of void spacing ($L/d \leq 10$), the deformation in the notch vicinity may be controlled by the high stress material response. Therefore, Fig. 16(b) shows the probability distribution for $K = 30$ MPa $\sqrt{m}$, re-calculated using the high stress values for $A_n$ and $n$ from Section 6.2.2. For comparison, the distribution for $K = 20$ MPa $\sqrt{m}$ from Fig. 16(a) is replotted in Fig. 12(b) (note the different x-axis range in the two figures). It is seen that when the high stress material properties are used, the distribution of failure times for $K = 30$ MPa $\sqrt{m}$ changes significantly, though the median initiation time is only slightly different (approximately 125 h). Note also that the probability of crack initiation for higher times is relatively low for this case—the high stress model predicts that there is a 20% probability that failure has not occurred within $2 \times 10^8$ h (failure probability is 80% at $t = 2 \times 10^8$ h). Thus the high stress model may overestimate survival probabilities for long times.

The above analysis has assumed that the macroscopic failure strain, $\varepsilon^c$, in Eq. (12), is independent of distance, $r$, in contrast to the result observed in Fig. 12(b). In order to include such dependence within a fracture mechanics-based framework, a comprehensive relationship which links the macroscale failure strain in the RR model, $\varepsilon^c$, and the void spacing, $L/d$, is required. The results presented here are a first step towards the development of a probabilistic microstructurally-based life prediction method for a single crystal alloy with low microvoid density, such as CMSX4. To include the effects of variability in material properties on the distribution of initiation times, e.g. tensile modulus, strain rate sensitivity (quantified by $n$ in the RR model), it is expected that a Monte Carlo-based approach would be needed, (see e.g. Nikbin et al., 2003).

As discussed in Section 6.2.3 diffusion and interdiffusion processes linked to notch surface oxidation alter the local phase composition of the material leading to the formation of a pure $\gamma$ region in the vicinity of the notch. The presence of this $\gamma$ region enhances the strain accumulation and results in a shorter predicted crack initiation time. For the commercial superalloy, CMSX4, under the conditions examined in this work, the effect of oxidation on the predicted life is small. This effect could be incorporated within the RR model presented here through the use of a macroscale failure strain, $\varepsilon^c$, which is dependent on the oxidation exposure time.

8. Conclusions

Coupled finite element diffusion–deformation analyses have been performed to study crack initiation in a notched compact tension specimen of a single crystal superalloy. The specimen contains a micro-void, representing a material casting defect, near the notch. Under mechanical loading, significant inelastic strain was shown to accumulate at the void surface. Using a strain-based failure criterion, implemented at the meso-scale, the time to crack initiation under creep loading was predicted.

As a result of the diffusion and interdiffusion processes linked to notch surface oxidation, the phase composition of the material is altered locally and introduces a pure $\gamma$
region in the notch area. The presence of the $\gamma$ region enhances the strain accumulation and leads to earlier crack initiation.

The model predicts that the crack initiation time is strongly dependent on the void location but is less sensitive to environment (i.e. whether oxidation occurs) for the cases examined. The weak effect of environment is due to the relatively low magnitude of the oxide growth rate constant, $\kappa_p$, for CMSX4 and the high applied loads considered in the analyses.

A probabilistic framework for the prediction of the time to crack initiation for the single crystal alloy has been proposed. The framework incorporates the strong sensitivity of the result to void location relative to a stress concentration such as a notch. The case of an assumed exponential distribution of void spacing has been examined and it has been shown that the predicted time to crack initiation can be obtained in closed form. Validation of the proposed approach from crack growth measurements in CMSX4 at high temperatures is ongoing.

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