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**In-situ** SEM study of slip-controlled short-crack growth in single-crystal nickel superalloy

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**A R T I C L E  I N F O**

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- Single crystal
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- Short-crack growth
- Crystal orientation
- Crack-growth rate

**A B S T R A C T**

Initiation and growth of short cracks in a nickel-based single crystal were studied by carrying out in-situ fatigue experiments within a scanning electron microscope (SEM). Specimens with two different crystallographic orientations, i.e., [001] and [111], were tested under load-controlled tension fatigue in vacuum. Slip-caused crack initiation was identified at room temperature while initiation of a mode-I crack was observed at 650 °C. Slip traces continuously developed ahead of the crack tip once initiated and acted as nuclei for early-stage crack growth at both room and high temperature (650 °C). These slip traces were caused by accumulated shear deformation of activated octahedral slip systems, which were specifically identified by analysing the surface slip traces and crack-propagation planes. The crack-growth rates were evaluated against stress intensity factor range, revealing the anomaly of slip-controlled short-crack growth. The effects of crystallographic orientations and temperature on fatigue crack growth were subsequently analysed and discussed, including the influence of microstructural features such as carbides and pores.

1. Introduction

Nickel-based superalloys are predominantly used to produce turbine blades and discs in aircraft engines and land-based gas turbines [1]. Such components are generally subjected to low-cycle fatigue during start-up, normal operation and shut-down of the system. Fatigue-life prediction of turbine components is generally based on a “damage tolerance” approach, which requires a good understanding of crack initiation and propagation behaviours of the material under fatigue. During fatigue loading, dislocations multiply and accumulate within the material, resulting in significantly increased levels of their density. These dislocations arrange themselves in configurations with the lowest energy by forming persistent slip bands (PSBs), which are precursors for crack initiation. In precipitation-hardened materials, such as nickel-based superalloys, PSBs form as dislocations cut through the γ matrix and γ′ precipitates in a planar slip manner.

Small fatigue cracks that are formed immediately after initiation tend to exhibit anomalously high and irregular growth rates when compared to large cracks at a similar stress-intensity-factor range ΔK. Short cracks can also grow below the threshold AK found for large cracks [2]. It is known that at the same stress intensity factor, short cracks grow much faster, with a lower nominal threshold than long cracks. Propagation of short cracks depends significantly on local microstructural features such as grain boundaries, secondary phases and inclusions, particularly for polycrystalline materials [3]. As reported by Toh and Rainforth [4] for a polycrystalline nickel alloy (Waspaloy), short fatigue cracks generally propagated along planar slip bands, with fluctuations in crack growth rate. This was associated with such microstructural features as grain and twin boundaries, both of which acted as barriers in the early stage of crack growth.

It is also more challenging to evaluate growth behaviour of short fatigue cracks in single-crystal alloys because of its strong dependency on crystallographic orientations [1,5–7]. Different slip systems may be activated for different crystallographic orientations and testing conditions, strongly affecting crack-growth behaviour [6,8]. To date, crystallography-dependent failure behaviour is not fully understood for single crystal alloys, subjected to fatigue loading. Antolovich et al. [9] investigated fatigue-crack propagation in single-crystal CMSX-2 at 25 °C and 700 °C. At 25 °C, the crack growth was purely crystallographic and occurred exclusively along (111) planes. At 700 °C, both mode-I (normal to the loading direction) and octahedral crack growth were observed. Sengupta and Putatunda [10] studied the near-threshold fatigue-crack growth behaviour of CMSX4 at 20 °C, 650 °C and 800 °C, and observed a zig-zag crack growth along (111) planes at 20 °C but not at higher temperatures where the fracture surfaces were smooth and flat. Ma et al. [11] studied the effect of temperature on low-cycle-
fatigue (LCF) crack growth behaviour with smooth cylindrical specimens of a single-crystal nickel-based superalloy. Crystallographic cracking on one or several (111) planes was predominant at a lower temperature, whereas at higher temperatures the crack growth was confined to the γ-matrix and γ/γ′-interfaces.

Experimental evidence of “anomalous” short-crack growth in single-crystal alloys is relatively limited due to the absence of grain boundaries and grain orientation mismatch, which can result in multiple slip trace activity and promote crack growth along intersecting slip planes in polycrystal alloys [12]. By using in-situ SEM tests, Ma et al. [13] studied the LCF crack growth in a single crystal nickel-based superalloy at 550 °C, with the consideration of three different crystallographic orientations. In-situ observations show that carbides presented in the microstructure influenced the crack path and crack growth rate. Ma and Shi [3] studied behaviour of small fatigue cracks in a directionally solidified nickel-based superalloy DZ4 using in-situ SEM at 25 °C, 350 °C and 700 °C. The fatigue-crack growth occurred preferentially along slip bands at 25 °C and 350 °C but in mode I at 700 °C. The anomalous small-crack growth was found to be pronounced at 25 °C and 350 °C due to the effect of microstructural features such as carbides and pores. The growth rate of fatigue cracks generally increased with temperature and became less microstructure-dependent at 700 °C. Zhang et al. [14] investigated growth behaviour of small fatigue cracks in a nickel-base single-crystal alloy using in-situ SEM tests, and found that the conventional Paris law was no longer capable to characterise the crystallographic crack-propagation behaviour. By reconstructing crack-tip stress fields and calculating the resolved shear stresses in each octahedral slip systems using finite-element analysis (FEA), they found that maximum resolved shear-stress intensity parameter could be used to characterise the growth behaviour of such small cracks.

The processes of initiation and propagation of small cracks in single-crystal nickel alloys still need further studies in order to gain a better understanding of damage evolution and tolerance of such materials, thereby ensuring the structural integrity of components. In this paper, initiation and growth of fatigue cracks in a nickel-based single-crystal superalloy are investigated using in-situ SEM, with a focus on the effects of crystallographic orientation and temperature. The slip systems responsible for crack initiation and growth were identified with SEM observations as well as post-test EBSD analysis for both [001] and [111] orientations. The effect of microstructural features such as carbides and pores on fatigue-crack growth was also discussed. The crack growth rate was calculated based on post-test measurement and correlated with a range of a stress intensity factor, which revealed abnormal crack-growth behaviour as a result of a slip-controlled crack-growth process at an early stage.

2. Material and specimens

2.1. Material description and specimen preparation

The material studied was a nickel-based single-crystal (NBSX) superalloy MD2, with a chemical composition Ni-5.1Co-6.0Ta-8.0Cr-8.1W-5.0Al-1.3Ti-2.1Mo-0.1Hf-0.1Si (in wt%). The alloy MD2 is in typical FCC lattice structure, and all twelve octahedral slip systems are considered in this paper. Single-crystal rods with a defined orientation were manufactured employing a vacuum casting process. For this study, rods with two typical orientations – [001] and [111] – were produced by GE Power. The casted rods went through a solution heat treatment (8 h at 1285°C) and a two-stage precipitation hardening treatment (1080°C for 6 h and 870°C for 16 h) in argon and vacuum. Four dogbone-shaped specimens with a gauge length of 5 mm and cross section of 2 × 1 mm² (Fig. 1) were machined from single-crystal rods using an electrical discharge machining (EDM) process. A U-shape notch was also machined in the middle section of the specimen, with a depth of 0.4 mm and a notch-end radius of 0.16 mm (Fig. 1). Two specimens were prepared for analysis for each loading-axis orientation in [001] and [111]. The specimen surfaces were mechanically ground with 240, 400, 600, 1200 and 2000 grit sizes, and then polished with a diamond spray colloidal silica of abrasive size of 0.25 μm to achieve a surface finish of Rₛ = 20 nm. The polished surfaces of the samples were then etched in an aqueous solution of 4 g CuSO₄ + 20 ml HCl + 20 ml H₂O at room temperature prior to fatigue tests in order to reveal the prevailing microstructure for in-situ observations with SEM.

2.2. EBSD analysis of specimens

To determine the secondary crystallographic orientation (i.e., orientation of the specimen surface), electron backscattering diffraction (EBSD) analysis was conducted on the surface next to the notch root using JSM-7800F Field Emission Scanning Electron Microscope (FE-SEM) and HKL Tango software provided by Oxford Instruments. A step size of 1 μm was used during the EBSD analysis. After EBSD indexing, the three Euler angles (φ₁, φ₂, φ₃) were extracted for each sample and summarised in Table 1, together with the colour index and the inverse pole figure (IPF). These Euler angles were used to determine the activated octahedral slip systems which are responsible for the development of slip traces on the sample surface (Section 4.1).

3. Experimental procedure

In-situ fatigue tests were performed in the vacuum chamber of the SEM using a specially designed servo-hydraulic testing system, which provided precise control of cyclic loading. This setup can provide pulsating (sinusoidal wave) loads with a ± 1 kN capacity and a displacement range of ± 25 mm. Fatigue tests were carried out in load-controlled mode at a load ratio of 0.1 and a loading frequency of 10 Hz (sinusoidal waveform). Throughout the tests, a constant maximal stress of 700 MPa was imposed for [001] oriented specimens while the maximal stress was increased to 850 MPa for [111] oriented specimens considering the significantly increased stiffness for that orientation. For both [001] and [111] oriented specimen, fatigue tests were performed...
at room temperature (RT) and 650°C. For high-temperature tests, the specimens were heated using resistance coils of the setup. Uniform heating of specimens was confirmed by the adjustable thermocouples attached to their gauge section. The temperature was controlled within ± 1°C of the nominal temperature for all tests. A signal from the SEM was directly transferred to a computer via a direct memory access type A/D converter, allowing 1280×960 frames to be recorded successively. The SEM was operated at an accelerating voltage of 15kV. The SEM images of samples with fatigue crack were captured in-situ at different stages by pausing the fatigue tests in order to capture the crack length for later measurements.

4. Results and discussion

4.1. In-situ observation of crack initiation and growth

The in-situ SEM observations of processes of fatigue crack initiation and propagation are selectively shown in Fig. 2 for [001]-oriented specimen, loaded up to failure. Overall, crack initiation and growth were closely associated with slip bands developed during fatigue loading. Development of multiple slip traces at the U-notch root due to local stress concentration is shown in Fig. 2a. These slip traces are inclined at either 140° or 108° to the loading direction. To determine the corresponding slip systems, the rotation matrix \( \mathbf{g} \), which connects the sample coordinates with crystal coordinates, was obtained by using the three Euler angles \((\varphi_1, \phi, \varphi_2)\) extracted from EBSD analysis (Table 1) and the following relation:

\[
\mathbf{g} = 
\begin{pmatrix}
\cos \varphi_1 \cos \varphi_2 & \sin \varphi_1 \cos \varphi_2 & \sin \varphi_2 \\
\sin \varphi_1 \sin \phi & \cos \varphi_1 \sin \phi & -\cos \phi \\
\sin \varphi_2 \cos \phi & \cos \varphi_2 \cos \phi & \sin \phi
\end{pmatrix}
\]  

(1)

Next, the loading direction in crystal coordinates can be worked out using the rotation matrix:

\[
L_c = \mathbf{g} L_s,
\]  

(2)

where \(L\) is a vector of the loading direction in the system of coordinates for the crystal \((L_c)\) and the specimen \((L_s)\).

The slip traces on the specimen surface corresponded to [111] slip planes, and the angle \(\alpha\) between a slip trace and the loading direction...
was calculated using the following equation [15]:
\[ \cos \beta = (n^* \times Z_c) \cdot L_c. \]
(3)
where \( n^* \) is the slip-plane normal to a slip system \( \varepsilon \), \( Z_c \) is the direction perpendicular to the specimen's surface in crystal coordinates. The calculated angle \( \alpha \) (or its supplementary angle) was used as a reference to identify the active [111] slip plane associated with the slip traces.

Finally, the slip direction was determined by identifying the slip system (out of 12 primary slip systems) with the highest Schmid factor \( SF \), based on the following equation [16]:
\[ SF = |(L_c \cdot n^*)(L_c \cdot l^*)| \]
(4)
where \( l^* \) is the slip direction for the slip system \( \varepsilon \).

Here, the active slip systems were determined as [T11] [0T1] and (1T1) [110] octahedral slip systems, corresponding to the two slip traces (146° and 108°) observed on the sample, respectively (as indicated in Fig. 2). However, as the cyclic loading continued, only one of these slip traces prevailed and developed into a crack at ~5903 cycles, while the others remained as slip traces (Fig. 2b). With the crack growth more slip traces were developed in front of the crack tip, and the crack path started to change direction, as also observed in Zhang et al. [14]. In addition, some secondary cracks started to initiate from the newly formed slip traces (111) [0T1] (Fig. 2c) and retarded the growth of the main crack (Fig. 2d). However, secondary cracks were unable to compete with the growth of the main crack, which seemed to follow a zig-zag path controlled by the development of slip traces near its tip (Fig. 2e). At the final stage of crack growth, large crack tip opening displacement (CTOD) was observed (Fig. 2f), leading to a significant increase in the crack growth rate and fracture.

4.2. Effect of orientation on crack initiation and growth

Typical process of crack initiation and evolution for [111]-oriented specimen tested at room temperature is illustrated in Fig. 3. The crack initiated from one single slip trace developed at the root of the U-notch at ~16,250 cycles (Fig. 3a). It propagated along a zig-zag path at the early period as shown in Fig. 3b, with the development of secondary cracking along other slip traces. Interestingly, after another 994 cycles, a significant secondary crack initiated from a slip trace developed in front of the crack tip (Fig. 3c) and evolved into a long crack as shown in Fig. 3d. When this crack grew up to a certain level, another crack started to initiate from a high plastic zone on the path of the previous crystallographic crack (Fig. 3e and f) and became dominant in the propagation process (Fig. 3g and h). This secondary crystallographic crack was affected by the development of slip traces and grew parallel to the previous cracking plane. Also, carbides affected the propagation process (Fig. 3h): crack bifurcated when blocked by carbides. Generally, the crack can either alter its growth direction or propagate through a particle, depending on the strength of carbide and local stress.

Fig. 3. In-situ observation of fatigue-crack initiation and growth for [111]-oriented specimen loaded at room temperature (vertical loading direction).
concentration. In this study, a change of growth path was noticed (Fig. 3h). With increasing loading cycles, the crack was attracted to carbides and micropores due to local stress concentration (Fig. 3i–l).

Compared to results for the [001]-oriented specimen, it was noted that fewer slip traces developed initially but with higher intensity. However, for both cases, the crack initiated from slip traces developed at the notch root. With an increase in loading cycles, the crack grew along slip traces developed ahead of its tip, resulting in a zig-zag path. The zig-zag crack character was more obvious for [111] orientation since the slip traces developed with stronger intensity in the material. The alloy also exhibited increased fracture surface roughness in [111]-orientated specimen than [001]-orientated specimen [17]. Besides, carbides and pores had an effect on crack growth at a later stage, resulting in crack branching and deflection, as early-stage crack growth was mainly controlled by the development of slip traces. It should be noted that the fatigue life for [111] orientation was significantly longer than that for [001] orientation, even under a higher level of stresses. This was associated with a higher modulus for [111] orientation, where specimens experience lower deformation under stress-controlled loading conditions. As reported by Shi et al. [18], fatigue life was significantly shorter for [111] orientation under strain-controlled loading condition, due to a high level of stress generated in the material as a result of its higher modulus.

4.3. Effect of temperature on crack initiation and growth

Both [001]- and [111]-oriented specimens were also tested to assess crack evolution at high temperature (650°C), and the results are selectively shown in Figs. 4 and 5. In comparison with the in situ SEM observations at room temperature, cracks initiated in a totally different mode at 650 °C: they started in a direction perpendicular to the loading axis, instead of along slip traces, at the notch root (Fig. 4a). This was essentially a mode-I cracking phenomena. However, the subsequent crack propagation followed the slip traces developed during the cyclic deformation (Fig. 4b). EBSD and crystallographic analyses showed that they were traces of $(111) [011]$ and $(111) [01¯1]$ slip systems, which were consistent with two of the active slip systems of the [001]-oriented specimen at room temperature. The octahedral cracking mode was also reported in PWA1484 superalloy by Telesman et al. [19]. At intermediate temperatures, the crack growth in the single-crystal superalloy occurred either on a single octahedral slip plane or on multiple slip planes, resulting in large crystallographic {111} facets on the fracture surface [11]. Although the crack switched direction as shown in Fig. 4c, its path was in the corresponding slip trace (Fig. 4d and e). A large CTOD was also observed (Fig. 4f) resulting in a sharp increase in the crack growth rate. A secondary crack initiated and propagated from a slip trace (Fig. 4g and h). Eventually, this led to fracture in a crystallographic mode along the $(111) [011]$ slip system.

For the [111]-oriented specimen tested at 650 °C, similar crack-
initiation behaviour was observed at the notch root, i.e., perpendicular to the loading axis in Mode-I (Fig. 5a). It propagated along a trace of the (T11) [0Ti] slip system (Fig. 5b). When the crack changed direction, it followed the (1T1) [10T] octahedral slip system (Fig. 5c and d). The crack path was affected by presence of carbides, with the crack growing either through or around them, depending on both the local stress concentration and carbide strength. In this case, the crack grew in Mode-I for a few microns and then returned to the octahedral plane, as observed in Fig. 5f. When the crystallographic cracking was blocked by carbides, transverse cracking was more efficient in increasing stress intensity than inclined cracks [14]. The crack continued to propagate along slip traces (Fig. 5f) after traversing the carbide inclusion. At the final stage of crack growth, a large CTOD was observed in the specimen (Fig. 5g). Furthermore, as shown in Fig. 5h, crack bifurcation occurred when the propagation path was blocked by carbides or attracted by micropores; however, the propagation mode was dominated by a crystallographic fracture process.

At high temperature, more octahedral slip systems were activated for [111] orientation due to the reduced critical resolved shear stress as well as higher stress level (i.e., 850 MPa), which facilitated the slip-controlled plastic deformation. Therefore, shorter fatigue life was observed for [111] orientation at high temperature. The mode-I crack...
initiation at high temperature might also be related to the activation of multiple slip systems near the notch root at the early stage of fatigue, due to the reduced critical resolved shear stress at increased temperature. However, as soon as a crack is developed, the slip at the crack tip will be switched to a single-slip mode as it is favored by the crack-tip stress field (i.e., along or close to the direction of maximum shear stress). This was similar to the crack growth at room temperature.

4.4. Correlating crack-growth rate with range of stress intensity factor

Based on the observed SEM images, the crack length was measured and recorded against the number of cycles. As the crack path was not straight, the measured crack length was measured as the projected length normal to the loading direction. Evaluation of crack length with a number of fatigue cycles for all four specimens is compared in Fig. 6. In the log-linear plot, a linear relationship was observed which corresponds to the observations in [20]. According to Frost and Dugdale [21], a simple exponential formula can describe the crack growth under

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Fig. 5. In-situ observation of fatigue-crack initiation and growth for [111]-oriented specimen at 650 °C (vertical loading direction).
constant amplitude loading:
\[ a = a_0 e^{KN}, \]
where \( a \) is the projected length (perpendicular to the loading axis), \( a_0 \) is the initial flaw length, \( a \) is a constant and \( N \) is the number of fatigue loading cycles. This relationship also holds in the small-crack growth regime for anisotropic single-crystal nickel-based superalloys as apparent from Fig. 5a.

The fatigue crack-growth rate was also correlated with the range of stress intensity factor (SIF). Here, the mode-I SIF range was calculated according to a single-edge-notched tension panel with finite width and given by
\[ \Delta K = \Delta \sigma \sqrt{a + b} F(a/W), \]
where \( \Delta \sigma \) is the stress range, \( a \) is the fatigue-crack length, \( b \) is the depth of the notch, and \( W \) is the width of the specimen. Let \( \xi = a/W \), so the function of \( F(\xi) = a/W \) can be defined by the empirical equation as [22]
\[ F(\xi) = 0.265(1-\xi)^4 + (0.857 + 0.265\xi)(1-\xi)^{3/2}. \]

The finite-element (FE) method can be used to evaluate computationally the level of SIF at the crack tip. Here, FE simulations were performed by replicating the model geometry and applied load used in in-situ tests. The material was assumed to be elastic with the Young's modulus of 200 GPa and the Poisson's ratio of 0.3. The SIF in the FE model was evaluated at the crack length corresponding to the experimentally assessed one. The difference between SIFs calculated with the empirical equations and FE simulations was within 5%, which verifies the reliability of the results calculated with Eqs. (3) and (4).

As shown in Fig. 6b, the fatigue-crack growth rates exhibit clear dependence on the crystallographic orientation and temperature, and a significant “short-crack anomaly” was found in all specimens. The crack growth was observed to decelerate first and then accelerate with an increase in SIF. Apparently, the fatigue-crack growth rate (FCGR) shows a large scatter, common for a small-crack growth in single crystals and deviates from the class Paris's law [23]. At room temperature, the [001]-oriented specimen showed a higher FCGR than the [111]-oriented specimen, although the applied maximal stress for the [001] orientation (700 MPa) was lower than that for the [111]-oriented specimen (850 MPa), which can be attributed to a lower yield stress and tensile strength in the [001] orientation when compared to the [111] orientation. On the other hand, at 650 °C, the FCGR for the [001] orientation was higher than that for the [111] orientation, but the difference was less significant than that at room temperature. For the same crystallographic orientation, the alloy showed a greater FCGR at 650 °C than at room temperature. Crack-closure behaviour of these specimens may be partly responsible for the differences in the FCGR, as crack surfaces were much rougher for the [111] orientation, leading to an increased crack-closure effect.

5. Conclusions

An in-situ SEM study of fatigue-crack initiation and propagation was performed for a single-crystal nickel-based superalloy, focusing on the effects of orientation and temperature. At room temperature, deformation by crystallographic slip was responsible for crack initiation, while at 650 °C, the mode-I type fracture indicated the onset of cracking at the notch root. An early-stage crack growth followed slip traces developed during fatigue loading, irrespective of orientation and temperature. The slip traces were caused by shear deformation along octahedral slip systems. The crack growth rate showed a large scatter when related with the stress-intensity-factor range. Local microstructural inhomogeneities such as the carbides and pores were found to alter the propagation path as well as decrease the crack-growth rate, especially at the final stage of crack growth. This study has significant implications for damage-tolerance assessment of critical gas turbine discs and blades made of nickel-based single-crystal superalloys, e.g. for optimisation of their crystallographic orientation with respect to the loading axis for improved fatigue resilience via component design.

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