EFFECT OF MULTIAXIAL STRESS CONDITIONS ON MICROSTRUCTURE DEGRADATION OF NICKEL BASE SINGLE CRYSTAL SUPERALLOY CMSX-4 DURING CREEP

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Abstract

The effect of multiaxial stress conditions caused by a notch effect on microstructure degradation of single crystal nickel base superalloy CMSX-4 was studied during creep at a temperature of 950 °C, constant nominal stress of 120 MPa for 500 and 1000 h. Flat specimens with “U-type” of notch for tensile creep experiments were designed by elastic finite element method (FEM) calculations. FEM elastic-plastic analysis was performed to simulate creep of the notched specimens. Material creep behaviour was integrated into FEM calculations by user-defined subroutine using equations derived from experimentally measured data for cylindrical creep specimens tested at various constant nominal stresses. Stress relaxation during creep was taken into account during these calculations. Magnitude, distribution and orientation of stresses resulting from the specimen loading were calculated and their effect on microstructure degradation was described. Microstructure analysis of the crept specimens showed that notches affect significantly degradation processes of the studied superalloy. It is shown that the morphology of the γ’ precipitates within the notch affected regions depends on the magnitudes and orientation of calculated stresses.

Keywords: Nickel base superalloy, microstructure degradation, multiaxial stress state, creep, rafting

1. INTRODUCTION

Nickel base superalloys are widely used in aircraft and power engineering due to their excellent properties at high temperatures such as creep and oxidation resistance. A typical microstructure of these alloys usually contains L1\textsubscript{2}-ordered γ’ (Ni\textsubscript{3}(Al, Ti)) precipitates coherently embedded in γ (Ni base solid solution with face-centered cubic crystal structure) matrix. The mechanical properties of nickel base superalloys depend on the volume fraction, distribution, size and morphology of γ’ precipitates. At high temperatures, initial cuboidal γ/γ’ microstructure undergoes first the process of Ostwald ripening \cite{1, 2} and secondly the process of formation of spontaneous rafts \cite{3}. When external stress is applied in a direction parallel to [001] crystallographic direction, the cuboidal γ’ precipitates undergo directional coarsening (rafting) with the rafts oriented in a direction perpendicular to the loading axis. Formation of creep rafts has been intensively studied and relatively well described by many authors \cite{4-6}.

The first stage blades for industrial gas turbines or aircraft engines are often cooled with internal channels that act as notches during their service. However, there is lack of available data about the effect of multiaxial stress conditions caused by such notches on microstructure degradation of single crystal nickel base superalloys \cite{7}.
The aim of the present work is to study the effect of multiaxial stress conditions caused by a notch on microstructure degradation changes of single crystal nickel base superalloy CMSX-4. The CMSX-4 is a cast nickel base superalloy, which is mainly used in manufacturing single crystal high-pressure turbine blades for aircraft engines and stationary gas turbines for power engineering.

2. EXPERIMENTAL PROCEDURES

The alloy CMSX-4 with the chemical composition Ni-7.0Cr-9.0Co-0.6Mo-6.0W-7.0Ta-3.0Re-5.6Al-1.0Ti-0.1Hf (wt.%) was provided by ALSTOM Ltd. in the form of cylindrical heat treated single crystal bars with a diameter of 20 mm and a length of 190 mm. The bars were cut to smaller pieces using spark machining and then ground to rectangular cuboids with dimensions of 6x15x80 mm. Elastic finite element method (FEM) calculations were performed to design geometry of a flat creep specimen for the study of the effect of multiaxial stress conditions on microstructure degradation of single crystal nickel base superalloys during creep. Geometry of a flat creep specimen was designed with “U-type” of notch characterized by radius of 1.5 mm. Threaded heads of the creep specimens were lathe machined and final geometry and defined dimensions were achieved by precise grinding. Fig. 1a shows a notched CMSX-4 specimen before creep testing. Assuming service conditions of single crystal turbine blades, creep experiments were performed at a temperature of 950 °C, a constant nominal tensile stress of 120 MPa for 500 and 1000 h. In order to minimize surface oxidation, a special apparatus enabling creep under flow of a protective atmosphere (Ar or He) was constructed. Fig. 1b shows the arrangement of the apparatus schematically.

Microstructural analysis was performed by light optical microscopy (OM) and scanning electron microscopy (SEM). Samples for OM and SEM were prepared using standard metallographic techniques and etched in a reagent of 12.5 ml alcohol, 12.5 ml HNO₃ and 13.5 ml HCl.

In order to simulate creep within the notched specimens, FEM elastic-plastic analysis was performed. Magnitude, distribution and orientation of stresses resulting from the specimen loading were calculated using mesh with 3D 20-node quadratic elements. Creep behaviour of CMSX-4, necessary for FEM simulation, was integrated by user-defined subroutine using equation based on experimentally measured creep data of cylindrical specimens without notch tested at constant nominal stresses of 90, 120 and 150 MPa [8]. Stress relaxation...
during creep was taken into account for FEM calculations.

3. RESULTS AND DISCUSSION

Microstructure within the creep specimen at the start of the experiment is shown in Fig. 2. From the metallographic analysis it is clear that microstructure after the heat treatments consists of cuboidal shaped \( \gamma' \) precipitates embedded in \( \gamma \) matrix. Mean precipitate size and average volume fraction of \( \gamma' \) precipitates was measured to be \( 310 \pm 6 \) nm and \( 69.5 \pm 1 \) vol.\%, respectively. This is in agreement with previous measurements made by Lapin et al. [1].

Using SEM, three different regions of \( \gamma/\gamma' \) microstructure in the vicinity of notch after 1000 h of creep at constant nominal stress of 120 MPa at 950 °C were found: (i) region with only cuboidal \( \gamma' \) precipitates (region A), (ii) region with non-uniformly rafted \( \gamma' \) precipitates (region B) and (iii) region with uniformly rafted \( \gamma' \) (region C). Figs. 3a and 3b show microstructures related to region with non-uniformly and uniformly rafted \( \gamma' \) precipitates, respectively.

In order to simulate the effect of creep on geometry of flat specimens, elastic-plastic FEM analysis was performed. Based on time-strain creep deformation curves measured independently on cylindrical specimens without any notches at constant external nominal stresses \( \sigma_{\text{ext}} \) of 90, 120 and 150 MPa, creep equation specifying time dependency of strain for a wide range of stresses was determined and consequently used for creep user-souboutine preprogramming. Please note that negligible amount of strain was assigned to the lowest possible stress of 1 MPa within the loaded specimen (based on the FEM elastic analysis) to preclude negative values of strain for low stresses. General form of this fourth grade polynomial equation is as follows:

\[
\varepsilon_{\text{calc}} = a + bt + ct^2 + dt^3 + et^{0.5}
\]  

where \( \varepsilon_{\text{calc}} \) represents calculated strain, \( t \) is time and \( a, b, c, d \) and \( e \) represent numerical constants, whereas these are all functions of \( \sigma_{\text{ext}} \).
After derivation of Eq. (1), one can get formula for the calculated strain rate \( \dot{\varepsilon}_{\text{calc}} \):

\[
\dot{\varepsilon}_{\text{calc}} = b_r + c_r t + d_r t^2 + e_r t^{-0.5} \frac{\varepsilon}{2}
\] (2)

where \( b_r, c_r, d_r \) and \( e_r \) represent numerical constants. Eq. (1) makes it possible to calculate creep strains at a temperature of 950 °C and maximal external nominal stress of 200 MPa applied for up to 2000 h relatively accurately. Fig. 4 shows comparison between the experimentally measured (symbols) and calculated values (dashed lines) of strain for constant nominal external stresses of 90, 120 and 150 MPa during creep at 950 °C for 2000 h. Verification of Eq. (1) was done by plotting strain versus strain rate of calculated values (using Eq. (2)) against those experimentally measured. It is clear that both, time-strain and strain-strain rate behaviors based on Eqs. (1) and (2) are in very good agreement with those measured experimentally.

Fig. 4. Comparison between the experimentally measured (symbols) and calculated values (dashed lines) for constant nominal external stresses of 90, 120 and 150 MPa during creep at 950 °C for 2000 h: (a) dependence strain on time and (b) dependence of strain rate on strain.

Fig. 5 shows the sketch of “U-type” of notch with dashed line representing the path for further examination of stress state and related microstructure, with initial and final point I and II, respectively. According to the Cartesian coordinate system applied for the creep specimen, this path lies at xy-plane close to the surface, where the biggest effect of stress state on microstructure degradation is expected. Please note that x-axis represents [001] crystallographic direction and also loading axis during creep of the specimen. xy-plane is equal to crystallographic plane which normal vector is perpendicular to the [001] crystallographic direction. After applying external load \( \sigma_{\text{ext}} \), stress state at every point of examined path can be characterized by three normal stresses \( \sigma_x, \sigma_y, \sigma_z \), and six shear stresses \( \tau_{xy}, \tau_{xz}, \tau_{yx}, \tau_{yz}, \tau_{zx}, \tau_{zy} \) or only by three principal stresses \( \sigma_1, \sigma_2, \sigma_3 \), whereas the principal stress directions are the unit vectors of the coordinate system in the case where no shear stress component is present.
In order to define distribution of stresses within the creep notched specimen just after loading, FEM elastic analysis was performed and Von Mises stresses \( \sigma_{VM} \) defined as

\[
\sigma_{VM} = \sqrt{\sigma_1^2 + \sigma_2^2 + \sigma_3^2 - \sigma_1 \sigma_2 - \sigma_2 \sigma_3 - \sigma_3 \sigma_1}
\]  

were calculated. Distribution of \( \sigma_{VM} \) in the vicinity of the notch at the start of the experiment resulting from the external load at 950 °C is shown in Fig. 6. It is clear that the highest values of \( \sigma_{VM} \) are located close to the surface at the center of notch (maximal value of \( \sigma_{VM} = 176 \) MPa is located at the surface). On the other hand, the lowest values of \( \sigma_{VM} \) are typical for the ends of notch.

As mentioned before, one of the possibilities to define stress state is to define three principal stresses \( \sigma_1, \sigma_2, \sigma_3 \), whereas following can be considered for a given point in space:

\[
\begin{align*}
\sigma_{\text{max}} &= \max(\sigma_1, \sigma_2, \sigma_3) \\
\sigma_{\text{min}} &= \min(\sigma_1, \sigma_2, \sigma_3) \\
\sigma_{\text{mid}} &= (\sigma_1 + \sigma_2 + \sigma_3) - \sigma_{\text{max}} - \sigma_{\text{min}}
\end{align*}
\]

where \( \sigma_{\text{max}} \) represents the highest tensile stress, \( \sigma_{\text{min}} \) the highest compressive stress (if there is no compressive stress, it represents the lowest value of tensile stress) and \( \sigma_{\text{mid}} \) is so called stress intensity.

Fig. 8 shows the evolution of calculated stresses \( \sigma_{\text{max}}, \sigma_{\text{min}} \) and \( \sigma_{\text{mid}} \) along the length \( l \) of examined path from an initial point I to a final point II placed at the edge and center of the notch, respectively, after 1000 h of creep. This figure also shows the behaviour of angle \( \varphi \) that represents an angle between direction of calculated \( \sigma_{\text{max}} \) and main x-axis clockwise in xy-plane. Diagram in this figure is divided into earlier mentioned regions A, B and C according to the morphology of \( \gamma/\gamma' \) microstructure observed along the examined path.
After 1000 h of creep, there is no significant degradation of cuboidal γ'/γ' microstructure within the region A. This region lies in the vicinity of examined path up to 0.68 mm from the point I. Based on microstructural analysis and taking into account calculated values of $\sigma_{\text{max}}$, $\sigma_{\text{min}}$, $\sigma_{\text{mid}}$ and $\varphi$, one can say that even after 1000 h of creep at 950 °C, the magnitude of maximal principal stress of about 60 MPa is not enough to degrade microstructure into rafted one. Microstructure remains cuboidal and coarsening known also as Ostwald ripening of the γ' precipitates takes place predominantly. This is in agreement with earlier quantitative measurements made by Lapin et al. [9] performed on cylindrical creep specimens with multiple gauge sections at four constant nominal external stresses of 60, 90, 120 and 150 MPa at a temperature of 950 °C.

Before analyzing region B, region C will be discussed because of less complicated stress state in comparison to region B. After 1000 h of creep, fully rafted γ'/γ' microstructure was observed within this region, whereas predominant orientation of γ' rafts was find to be perpendicular to an external loading axis. This is shown in Fig. 9 that represents stress state at the point II ($\sigma_{\text{max}} = 107$ MPa, $\sigma_{\text{mid}} = 10$ MPa, $\sigma_{\text{min}} = -2$ MPa, $\varphi = 0^\circ$, $l = 3$ mm) and related γ' rafts. At this point, formation of the rafts is affected primarily by $\sigma_{\text{max}}$, whereas the magnitudes of $\sigma_{\text{mid}}$ and $\sigma_{\text{min}}$ are negligible in comparison to magnitudes of $\sigma_{\text{max}}$. Within this region, there is no significant change in magnitudes of principal stresses. The only change that can affect evolution of directional coarsening of the γ' phase is therefore the change of angle $\varphi$, which continuously increases from the point II to the end of region C from $\varphi = 0$ to about $\varphi = 50^\circ$. Accordingly to known fundamentals of rafting process [4], directional coarsening of the γ' phase occurs in a direction perpendicular to the applied load in superalloys with negative misfit values (the case of the studied CMSX-4). However, this agrees only for close vicinity of the point II at examined path through investigated notch. Even orientation of $\sigma_{\text{max}}$ with $\varphi = 50^\circ$ is insufficient for the change of predominant orientation of general axis of rafts within region C.
Fig. 9. Stress state at the point II of the examined path: (a) Main Cartesian coordinate system with direction of external load $\sigma_{\text{ext}}$. (b) Elementary stress state cube representing the effect of principal stresses $\sigma_{\text{max}}$, $\sigma_{\text{min}}$ and $\sigma_{\text{mid}}$ on orientation of $\gamma'$ phase after 1000 h of creep. Filled plane in elementary stress state cube represents plane parallel to xy-plane.

Fig. 10 shows stress state at point X ($\sigma_{\text{max}} = 72$ MPa, $\sigma_{\text{mid}} = 1$ MPa, $\sigma_{\text{min}} = -16$ MPa, $\phi = 50^\circ$, $l = 1.1$ mm) placed within the region B and related to coarsened $\gamma'$ phase. At this point, formation of rafts is affected primarily by $\sigma_{\text{max}}$ which has the highest magnitude from all principal stresses, but there is also a contribution from $\sigma_{\text{min}}$ that cannot be neglected. Moreover, $\sigma_{\text{min}}$ has compressive character and hence a tendency to orientate directional coarsening of $\gamma'$ phase parallel to the direction of $\sigma_{\text{min}}$. This kind of stress state seconded by high angular dimension of $\phi$ results in $\gamma'$ rafts with several protrusions in directions parallel and perpendicular to the external loading axis. In contrast to region C, high angular dimension of $\phi$ plays a role in general orientation of the $\gamma'$ raft which is possible to define by an angle $\alpha$. It represents the angle between the general axis of $\gamma'$ raft in xy-plane and main y-axis. As can be seen from the sketch in Fig. 12, $\alpha$ takes value of about $40^\circ$ at the point X.

4. CONCLUSIONS

The investigation of the effect of multiaxial stress conditions caused by a notch effect on microstructure degradation of nickel base single crystal superalloy CMSX-4 during creep at 950 °C for 500 and 1000 h under an applied stress of 120 MPa suggests following conclusions:
1. There is no evidence of directional coarsening of the cuboidal $\gamma'/\gamma'$ microstructure within the region affected by maximal principal stress values up to 60 MPa, even if there is a high magnitude of $\varphi$. Microstructure remains cuboidal, whereas the process of coarsening of the cuboidal $\gamma'$ precipitates known as Ostwald ripening takes place within this region predominantly.

2. Fully rafted $\gamma'/\gamma'$ microstructure with the rafts oriented perpendicularly to the [001] crystallographic direction were observed within the region affected primarily by $\sigma_{\text{max}}$, where $\sigma_{\text{mid}}$ and $\sigma_{\text{min}}$ have negligible magnitudes. Even at $\sigma_{\text{max}}$ with high angular magnitude of $\varphi$, the main axis of directionally coarsened $\gamma'$ phase remains perpendicular to the [001] crystallographic direction, what is in contrast to the known theories of rafting.

3. Non-uniform directional coarsening of the $\gamma'$ phase was observed within the region, where beside the effect of $\sigma_{\text{max}}$, the effect of non-negligible $\sigma_{\text{min}}$ with compressive character should be also taken into account. This kind of stress state seconded by high magnitude of $\varphi$ results in a directional coarsening of the $\gamma'$ phase with several protrusions in directions parallel and perpendicular to the [001] crystallographic direction. Moreover, in contrast to the region where only $\sigma_{\text{max}}$ was affecting degradation of microstructure, high values of $\varphi$ affects orientation of the $\gamma'$ rafts.

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