Abstract
The effect of uniaxial and multiaxial stress conditions on microstructure degradation of single crystal nickel based superalloy CMSX-4 was studied during creep. Cylindrical specimens with multi gauge sections for tensile creep at uniaxial stress conditions and “U type” of notch for creep at multiaxial stress conditions were designed by elastic finite element method (FEM) calculations. The width of the γ' (Ni3(Al,Ti)) rafts decreases and length of the γ' rafts and width of the γ channels increase with increasing applied stress and creep time. The multiaxial stress conditions affect the degradation process of the γ/γ' microstructure when compared to that observed at uniaxial stress conditions. The observed degraded microstructure of creep specimens tested at laboratory conditions is related to that of ex-service turbine blades. External stress of about 50 MPa resulting from centrifugal forces during blade service is determined to be critical, beyond which the role of the internal stresses in formation of the rafted microstructure can be neglected. This stress is in agreement with the creep experiments at a constant nominal stress of 60 MPa which showed classically rafted microstructure with minor fraction of spontaneous rafts. The width of the γ' and γ phases increases with increasing temperature and decreasing stress. Increased width of the γ' phase in regions with lower stresses is in agreement with the previous laboratory experiments, wherein the widest γ' rafts were measured in specimens exposed to the lowest experimental stresses in later stages of creep.

Key words: Nickel based superalloy; Creep; Notch effect; Microstructure degradation; Turbine blade.

INTRODUCTION
Single crystal nickel based superalloys have been developed for processing of turbine blades operating at high temperatures and complex stresses in stationary gas turbines and aircraft engines. The main requirements on these materials are: (i) resistance against complex damage mechanisms during mechanical loading at high temperatures, (ii) resistance against corrosive effects of hot gases and (iii) optimized balance of mechanical and technological properties. Due to strategic importance of nickel based superalloys, particularly in the aircraft industry, superalloys have become one of the most studied groups of metallic materials [1-6]. A typical microstructure of these alloys usually consists of L12-ordered γ' (Ni3(Al,Ti)) precipitates coherently embedded in γ (Ni based solid solution with face-centered cubic crystal structure) matrix. The mechanical properties of nickel based 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 [7] and consequently the process of formation of spontaneous rafts [8]. When external tensile 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, e.g. [9-10]. 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 a lack of available data about the effect of multiaxial stress conditions caused by such notches on microstructure degradation of single crystal nickel based superalloys [11].

The aim of the present work is to study the effect of uniaxial and multiaxial stress conditions caused by notches on microstructure degradation changes of single crystal nickel based superalloy CMSX-4 during creep. In addition, microstructure degradation of ex-service single crystal turbine blades is presented.
EXPERIMENTAL PROCEDURE

As-cast ingot of nickel base superalloy CMSX-4 with diameter of 80 mm, length of 250 mm and chemical composition Ni-6.5Cr-9.0Co-0.6Mo-6.0W-6.5Ta-3.0Re-5.6Al-1.0Ti-0.1Hf (wt.%) was supplied by Canon-Muskegon (USA). The ingot was cut to smaller rectangular rods by electro-spark machining and lathe-machined to cylindrical rods with a diameter of 12 mm and length of 120 mm. Single crystals with [001] crystallographic orientation were prepared by directional solidification at a temperature gradient in liquid at the solid-liquid interface of 1x10^4 °C/m and growth rate of 2.78x10^5 ms^{-1} in a modified Bridgman type apparatus. Crystallographic orientation of directionally solidified bars was controlled by Laue X-ray diffraction technique. Maximum deviation from <001> crystallographic direction was measured to be 7 degrees. The single crystal bars were subjected to solution annealing at 1315 °C for 6 h in high purity argon atmosphere which was followed by a rapid cooling to room temperature in flowing argon. The heat treatment was finalised by two steps precipitation hardening at 1140 °C for 2 h and 870 °C for 20 h in air followed by gas fan cooling to room temperature.

The specimens for creep at uniaxial and multiaxial stress conditions were prepared by lathe machining. The microstructure degradation during uniaxial creep was studied at a temperature of 950 °C, applied stresses of 60, 90, 120 and 150 MPa for various time up to 2000 h using cylindrical creep specimens with multiple gauge sections. Creep specimens for the study of the effect of multiaxial stress conditions on microstructure degradation were designed with "U-type" of notch using finite element method (FEM) calculations.

Ex-service single crystal turbine blades from CMSX-4 superalloy after 12,500 h service were provided by Siemens. These blades were cut to smaller pieces by electro-spark machining and subjected to microstructure evaluation. The observed microstructures were related to local stresses and temperatures within the turbine blade.

Microstructural analysis was performed by optical microscopy (OM) and scanning electron microscopy (SEM). OM and SEM samples were prepared by grinding on abrasive papers, polished on diamond pastes up to a grain size of 0.25 μm and etched in a reagent of 12.5 ml alcohol, 12.5 ml HNO₃ and 13.5 ml HCl. Quantitative metallography was performed on digitalized micrographs using a computerized image analyser. Five basic microstructural parameters were evaluated in the specimens: (i) size of cuboidal γ' precipitates, (ii) volume fraction of γ' precipitates, (iii) width of γ channels, (iv) width of γ' rafts and (v) length of γ' rafts on longitudinal sections of creep specimens. All measured values were evaluated statistically using appropriate distribution functions.

RESULTS AND DISCUSSION

3.1 Microstructure degradation during uniaxial stress conditions

Fig. 1 shows the initial microstructure of the creep specimens prepared from single crystal bars of CMSX-4 superalloy. The microstructure consists of cuboidal shaped γ' precipitates embedded in the γ matrix. Mean size and average volume fraction of the γ' precipitates is measured to be (310 ± 6) nm and (69.5 ± 1) vol.%, respectively.

During creep at 950 °C and applied stresses ranging from 60 to 150 MPa coarsening and rafting of the cuboidal γ' precipitates is observed, as seen in Fig. 2. The well developed γ' rafts separated by the γ channels are oriented nearly perpendicularly to the loading direction which is parallel to the [001] crystallographic direction of the creep specimen. Nabarro et al. [9] showed that the driving force for development and orientation of the γ' rafts is: (i) the lattice misfit between γ' precipitates and γ matrix, (ii) elastic...
Fig. 2. Evolution of the γ'/γ microstructure within the creep specimen tested at uniaxial stress conditions, temperature of 950 °C, creep time of 500 h and applied stresses of 60, 90, 120 and 150 MPa.

Fig. 3. Variation of measured microstructural parameters of the γ'/γ microstructure with the applied stress after uniaxial creep at 950 °C: (a) width of the γ' rafts, (b) length of the γ' rafts, (c) width of the γ channels. The creep time is indicated in the figures.
mismatch between the γ’ precipitates and the γ matrix and (iii) level of applied external stresses. Three basic microstructural parameters were measured for the rafted microstructure: (i) width of the γ channels in a direction perpendicular to the γ’ rafts, (ii) width of the γ’ rafts and (iii) length of the γ’ rafts on longitudinal sections of the creep specimens. All measured values are evaluated statistically using appropriate distribution functions. Fig. 3 shows variation of the microstructural parameters of degraded γ/γ’ microstructure with the applied stress after creep testing for various time. The values of the width of the γ’ rafts in specimens tested at 60 and 90 MPa for 100 h are affected by a transient type of microstructure between cuboidal and rafted one. For the degraded microstructure with well developed rafts, the width of the γ’ rafts decreases (Fig. 3a) and their length increases (Fig. 3b) with increasing applied stress and creep time. The width of the γ channels increases with increasing applied stress and creep time, as seen in Fig. 3c.

3.2 Microstructure degradation during multiaxial stress conditions

Fig. 4 shows the notched tensile creep specimen with "U" type of notch tested at 950°C and nominal stress in the notched region of 90 MPa for 1000 h. After 1000 h of creep, there is no significant degradation of the cuboidal γ/γ’ microstructure in the regions which are not affected by the notch effect. The coarsening known also as Ostwald ripening of the cuboidal γ’ precipitates takes place predominantly in the regions subjected to uniaxial stress conditions. This is in agreement with the quantitative measurements performed on the cylindrical creep specimens with multiple gauge sections at four constant applied stresses. On the other hand, the microstructure in the region affected by the "U" notch is well rafted. The rafts are well oriented at the angle of 90° to the tensile axis in the central region of the specimen. The microstructure in the vicinity of the notch is not regular. The γ’ rafts are oriented in two main directions: (i) nearly perpendicularly to the tensile axis and (ii) nearly parallel to the tensile axis. As shown by Gebura and Lapin [11] and Serin et al. [1], rafting of the γ’ precipitates during high temperature low stress creep of single crystal superalloys depends on the stress state and stress level. The FEM calculations of stress distribution in the notch affected region performed by Gebura and Lapin [11] showed that there is no evidence of directional coarsening of the cuboidal γ/γ’ microstructure within the region affected by maximal principal stresses up to 60 MPa, even if there...
is a high magnitude of angle $\phi$ between direction of calculated maximal principal stress and main $x$-axis clockwise in $xy$-plane. Fully rafted $\gamma/\gamma'$ microstructure with the rafts oriented perpendicularly to the [001] crystallographic direction are observed within the region affected primarily by maximal principal stress, where the stress intensity and compressive stresses have negligible magnitudes. Non-uniform directional coarsening of the $\gamma'$ phase is observed within the region, where beside the effect of maximal principal stress, the compressive stresses contribute to the microstructure degradation. This stress distribution enhanced by high values of $\phi$ lead to 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 maximal principal stresses affect the degradation of microstructure, high values of $\phi$ affects orientation of the $\gamma'$ rafts.

3.3 Microstructure degradation of turbine blades

The examined single crystal turbine blades without internal cooling channels were manufactured by directional solidification (Fig. 5). The initial microstructure of the blades consisted of the cuboidal $\gamma'$ precipitates with a mean size of about 350 nm embedded in the $\gamma$ matrix. Fig. 6 shows different types of microstructure observed within the analysed blades. Figs. 6a and 6b show spontaneously rafted $\gamma/\gamma'$ microstructure in the vicinity of the airfoil tip operating at a temperature of about 980 °C and stress of about 30 MPa. The spontaneously rafted microstructure consists of the $\gamma'$ rafts oriented perpendicularly as well as parallel to the loading axis. The formation of spontaneous rafts in the CMSX-4 superalloy results from internal dendritic stresses [8]. Figs. 6c and 6d show well rafted microstructure formed in the middle part of the airfoil part operating at a temperature of about 915 °C and centrifugal stresses of about 110 MPa.

**Fig. 5.** Ex-service single crystal turbine blade from CMSX-4 alloy after service for 12,500 h.

**Fig. 6.** SEM micrographs showing different microstructures observed within the turbine blades after 12,500 h of service: (a) and (b) spontaneously rafted $\gamma/\gamma'$ microstructure; (c) and (d) well developed creep rafts; (e) well developed creep rafts in coexistence with semi-rafted microstructure; (f) cuboidal microstructure in coexistence with semi-rafted one.
Figs. 6e and 6f shows degraded microstructure of the turbine blade in the vicinity of root part operating at a temperature of about 810 °C and stresses of about 190 MPa. In this region, the microstructure consists mainly of coarsened cuboidal γ' precipitates and some isolated regions with γ' rafts in the γ matrix.

CONCLUSIONS
The study of the effect of uniaxial and multiaxial stress conditions on microstructure degradation of single crystal nickel based superalloy CMSX-4 during creep suggests the following conclusions:

1. The initial cuboidal γ/γ' microstructure of single crystal CMSX-4 superalloy is unstable during 950 °C creep at uniaxial stress conditions. The width of the γ'(Ni3(Al,Ti)) rafts decreases and length of the γ' rafts and width of the γ channels increase with increasing applied stress and creep time during tensile creep testing.

2. There is no evidence of directional coarsening of the cuboidal γ/γ' microstructure within the region affected by maximal principal stress values up to 60 MPa, even if there is a high magnitude of φ during creep at multiaxial stress conditions. Fully rafted microstructure with the rafts oriented perpendicularly to the [001] crystallographic direction is observed within the region affected primarily by maximal principal stresses, where the stress intensity and compressive stresses have negligible magnitudes. Non-uniform directional coarsening of the γ' phase is observed within the region, where beside the effect of maximal principal stresses, the non-negligible compressive stresses takes place.

3. The external stress of about 50 MPa resulting from centrifugal forces during blade service is determined to be critical for formation of spontaneously rafted microstructure. Higher stresses leads to fully rafted or partially rafted microstructure depending on local temperature within the turbine blade.

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REFERENCES