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Investigation of localized damage in single crystals subjected to thermalmechanical fatigue (TMF)

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Abstract

The deformation and damage mechanisms arising during thermalmechanical fatigue (TMF) of a CMSX-4 and high-Cr single crystal superalloy, SCA425 have been investigated and a completely new failure mechanism involving recrystallization and oxidation has been discovered. The primary deformation mechanism is slip along the {111} planes. The deformation is highly localised to a number of bands, where recrystallization eventually occur during the thermalmechanical fatigue process. When the final failure occurs along these recrystallized bands it is accompanied by the formation of voids due to the presence of grain boundaries. The damage process is further enhanced by oxidation, since recrystallization occurs more easily in the J‘ depleted zone under the oxide scale. The macroscopic as well as the microscopic damage and fracture mechanisms are varying with alloy and heat treatment. The aim of this work is to further investigate, discuss the localisation of damage into twins and extremely localized rafted deformation bands.

Keywords: Thermalmechanical fatigue; Deformation mechanisms; Recrystallization; Single crystal superalloy; Rafting

1. Introduction

Thermalmechanical fatigue (TMF) in superalloys is growing in importance since changes in demand, and competition within the power generation market force the power plants in many countries to operate under cyclic conditions. Very recent work has shown that the deformation and damage mechanisms in low-Cr single crystal superalloys during TMF are very different from those traditionally reported to occur under creep or isothermal fatigue [1-3]. Unfortunately, at this stage relatively little is known about the influence of alloy composition on the mechanisms active during thermalmechanical fatigue of single crystal superalloys. In a previous study on CMSX-4 [1] it was found that highly localised twinning is the main deformation mechanism operating during out-of-phase thermalmechanical cycling and that the twins are extended across the complete cross-section of the specimens tested. An example of results from the TMF testing is shown in Fig. 1 where almost the same TMF results in terms of life produced a completely different fracture due to long term ageing (Fig. 1b). The highly localized deformation was also found to promote the precipitation of µ-phase, but the associated recrystallization was probably the most remarkable observation. Recrystallization was observed within the deformation bands, with the interception of twins of different orientations acting as initiation sites for this process. It was further observed that crack propagation occurs rapidly along the twinning plane especially if recrystallization had also occurred. Second generation single crystal superalloys, e.g. CMSX-4, typically have excellent creep and good oxidation properties but are known to be sensitive to sulfidation and hydrogen embrittlement [4, 5]. For industrial gas turbine applications it is often desirable to use alloys with a higher corrosion resistance than what is typical for the second generation single crystals. SCA425 is a single crystal nickel-base superalloy with high chromium content that was developed in a European Brite-Euram program [6] with the aim to create a high corrosion resistance single crystal alloy suitable for industrial gas turbine applications. In this study, CMSX-4 and SCA425 in two slightly modified versions has been investigated with

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2. Experimental details

Compared to the original version of SCA425, the modified alloy in this study has additions of Hf and is therefore referred to as SCA425Hf where 2wt% Ti is replaced by 0.5wt% Al and 3wt% Ta in order to further increase oxidation resistance. The chemical composition for the main alloying elements is shown in Table 1 and a more detailed description of the heat treatment is found in [1, 7]. The specimens were machined from cast bars with their longitudinal axis parallel to the [001]-direction.

Out of phase (OP) thermal mechanical fatigue tests were conducted under mechanical strain control in the 100–950ºC, (50ºC lower than for CMSX-4 due to risk for oxidation) temperature range with a strain ratio of R = εmin /εmax = -0.1 by the use of an MTS 810 servo-hydraulic thermal mechanical fatigue machine with the MTS model 793 software. In order to achieve a stabilized mean stress early in the tests a 20 hour hold time was applied at the maximum temperature (Tmax) during the first cycle. For all subsequent cycles a 5 minute hold time was applied and the total cycle time was 777 seconds. The combination of R-ratio and longer hold time in the first cycle is believed to better represent the real situation for most engineering components under OP-TMF loading. The CMSX-4 material was TMF tested in a similar way but the maximum temperature was 1000 ºC and in two conditions called virgin and pre long term aged where 1000 ºC was applied for 4000h after initiating the test (see [1, 7] for more details).

After testing to failure, the ruptured fatigue specimens were sectioned and prepared for micro structural investigations. Sectioning was performed both perpendicular and parallel to the longitudinal axis as well as parallel to the fracture surface which in most cases was parallel to any of the \( \{111\} \) planes. The samples were prepared by grinding and mechanical polishing and analysed using an analytical scanning electron microscopy (SEM), Hitachi SU70 operating at 20 kV. No etching was performed on the samples and the contrast was achieved from differences in composition and crystallographic orientation only. Chemical composition information was obtained using an energy-dispersive X-ray system (EDS) and a wavelength dispersive spectrometer (WDS) from Oxford Instruments. Orientation imaging microscopy (OIM) was performed using an electron back-scattering diffraction (EBSD) system from HKL Technology.

### Table 1. Alloy composition

<table>
<thead>
<tr>
<th>Alloy/Wt%</th>
<th>Al</th>
<th>Co</th>
<th>Cr</th>
<th>Hf</th>
<th>Mo</th>
<th>Re</th>
<th>Ta</th>
<th>Ti</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>CMSX-4</td>
<td>5.65</td>
<td>9.6</td>
<td>6.4</td>
<td>0.11</td>
<td>0.61</td>
<td>2.9</td>
<td>6.6</td>
<td>1.02</td>
<td>6.4</td>
</tr>
<tr>
<td>SCA425Hf</td>
<td>4.0</td>
<td>4.9</td>
<td>16.2</td>
<td>0.4</td>
<td>1.0</td>
<td>4.9</td>
<td>2.04</td>
<td>4.0</td>
<td></td>
</tr>
<tr>
<td>SCA425+</td>
<td>4.52</td>
<td>5.03</td>
<td>15.6</td>
<td>0.1</td>
<td>1.0</td>
<td>7.95</td>
<td>4.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3. Results

More detailed results from the OP-TMF tests can be found in [1, 7]. In summary CMSX-4 is superior to the others despite the fact that it is tested at a higher maximum temperature. Comparing SCA425Hf and SCA425+ shows a higher creep relaxation resistance for SCA425Hf.

Fig. 1. Macrofractographs of CMSX-4 TMF specimens cycled to failure (a) virgin condition; (b) long term aged condition
The general fracture appearance of CMSX-4 is shown in Fig. 1 as a remarkable difference between the long term aged and virgin specimens. The other alloys show in case of SCA425Hf crystallographic type while the SCA425+ tend to be ductile like the aged CMSX-4. The fracture surface is purely crystallographic and fracture occurs along one of the \{111\} planes. Parallel to the mirror like fracture surface one can also identify bands of localized deformation with the same appearance as the bands seen in Fig. 1b. Such a band can also be seen in the longitudinal cross section in Fig. 2(a), where a step has clearly formed on the specimen surface. The bands often extend through the entire cross section of the specimen, and it is noteworthy that one can see void formation often occurs along the bands inside the material, see Fig. 2(b).

3.1. Scanning electron microscopy

3.1.1. Virgin CMSX-4 material
The microstructure of the virgin un-tested CMSX-4 material is shown in Fig. 3, and consists of cuboidal \(\gamma'\) precipitates orientated along the \(<001>\) direction in a matrix of \(\gamma\). Under the conditions examined and despite the relatively short duration of the TMF testing, rafting or coalescence and coarsening was found to occur rapidly.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig2.png}
\caption{(a) Polished cross section of SCA425Hf showing deformation band with a crack like defect shown in (b).}
\end{figure}

see for example Fig. 4 which corresponds to virgin material after TMF testing with \(\Delta e_{\text{mech}}=0.9\%\) and approximately 200 cycles. Rafting was much more pronounced for the sections parallel to the longitudinal direction of the specimen. Since the specimens are loaded in compression at high temperature during OP-TMF testing rafting along the tensile, \([001]\) direction is consistent with the observations made during creep where it has been reported for CMSX-4 - and other alloys with a negative \(\gamma/\gamma'\) misfit - the rafts in tensile-loaded creep specimens are perpendicular to the stress axis, while the rafts in compressively-loaded specimens are parallel to the stress axis [8-10].

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig3.png}
\caption{Secondary electron image of CMSX-4 showing typical microstructure of virgin un-tested material.}
\end{figure}

As seen in Fig. 5(a), the deformation due to the thermalmechanical fatigue loading was found to be localized; bands of deformation which extended across the complete width of the cross-section of the specimens were displayed. With respect to the
axial direction of the specimens an angle of approximately 55° was determined for the normal direction of the planes along which these slip bands are active.

![Fig. 4 Backscatter electron micrograph showing microstructure of TMF-cycled virgin material: (a) longitudinal section (b) transverse section.](image)

These bands of localized deformation thus extend on the \{111\} planes of the single crystal. Within these bands, small precipitates of TCP phases were commonly observed, see Fig. 5(b). The precipitates were generally spherically shaped with a size between 0.1 and 0.5μm. WDS analysis revealed that the particles were rich in Ta, W and Re, consistent with CMSX-4's known propensity for precipitation of the μ-phase [11].

![Fig.5 Backscatter electron micrographs of TMF-damage with localized deformation appearing as bands decorated with precipitates (b).](image)

By using the EBSD technique one can determine that there is a 60° misorientation in the bands of localized deformation compared to the matrix, see Fig. 6, which confirms that micro-twinning has occurred. It has been found that the width of these twins can approach 2μm and that they often grow in parallel pairs.

For test pieces which have been cycled until failure, analysis confirms that the crack propagation occurs along the bands of localized deformation, see Fig. 7(a). The cracks propagate preferentially along one of the twin boundaries rather than within the twin itself. For example, in Fig. 7(b) a crack is imaged in which one has a band of localized deformation and TCP-phases on one side of the crack and only the single crystal matrix on the other side of the crack. Within the band of localized deformation close to the crack it also appears as that re-crystallization has occurred. That this is really the case is confirmed by the EBSD mapping in Fig. 8; the different colours or grey levels correspond to grains of different orientations. No signs of γ′ were seen within the individual recrystallized grains so it must be assumed that a microduplex structure has developed whereby each grain consists of either the γ- or γ′-phase. Even if recrystallization is always seen along the fatigue cracks, the presence of recrystallized grains is not limited to the crack front. Fig. 9 shows that a possible starting point for the recrystallization process is the intersection between twins growing in different directions. Since the twins in the virgin material grow along the complete cross-section of the
sample, intersecting twins are rather frequently observed. Fig. 9 shows that no preferred crystallographic texture is present in the recrystallized regions. The implications of these observations are discussed further.

Fig. 6 Crystallographic orientation map produced by EBSD with corresponding misorientation profile transverse to a band of localized deformation.

Fig. 7 (a) Backscatter electron micrographs showing cracking along a band of localized deformation. (b) Micrograph showing microstructure close to the crack, decorated with TCP phase and showing signs of recrystallization.

Fig. 8 Region within a band of localized deformation characterised by (a) SEM, (b) EBSD.
3.1.2. Aged CMSX-4 material

In the material aged at 1000ºC for 4000 hours, extensive development of TCP precipitates was found to have occurred but these are not homogeneously distributed within the material. The precipitation of TCP phases occurred mainly in the dendrite cores, as seen in the transverse section in Fig. 10(a), and this is consistent with the known partitioning behaviour of Re to the solid phase during solidification. This causes a “banded” microstructure along the longitudinal direction of the specimen, corresponding to the direction of solidification during casting, see Fig. 10(b).

It is believed that the TCP phases act as efficient obstacles for the propagation of the twins and associated slip bands, thus preventing the deformation from being as highly localised as for the virgin material, see Fig. 11. Consequently, the twins cannot extend across the complete cross-section of the specimen and therefore many more slip bands or twins are activated more homogeneously as compared to the virgin material. The width of the twins is also in general much smaller for the aged material compared to the virgin material. Also seen in Fig. 11(a) is a contrast pattern in the inter-dendritic region which is a consequence of a mis-orientation of certain regions. This is illustrated by the EBSD-map in Fig. 12. Almost no recrystallization was observed for the aged material but the misorientation profile show signs of intensive dislocation activity. The failure behaviour is also like what is shown in Fig. 2.
3.1.3. SCA 425

The SCA425Hf alloy showed clear slipbands on a macroscale and close to the crystallographic fracture surface those bands studied in SEM showed a rafted $\gamma'$ structure along the bands as shown in Fig. 13. Those bands are close to the fracture surface and around inclusions developed into recrystallized zones leading to development and growth of the final damage of the type shown in Fig. 1a. The thickness of the bands can be up to 5µm and they always propagate along one of the $\{111\}$ planes. Most of the bands, as the one shown in Fig. 13b, do not show any or little crystallographic misorientation [7] relative to the bulk material, even if massive shearing or slip has occurred. The thickness of the recrystallized zone is approximately 3µm and the sizes of the individual grains are in the range of 100-500 nm. The recrystallization is not restricted to the primary fracture surface. Other deformation bands parallel to the fracture surface also show at least partial recrystallization. A possible starting point for the nucleation of recrystallized grains is found when slip lines interfere with features in the microstructure such as carbides or topologically close packed (TCP) phases, see [7]. When the recrystallization has started, it continues along the slip lines where the stored energy from the plastic deformation is high. Along these recrystallized slip bands one can often observe void formation. Another observation made during the present investigation was that the $\gamma'$-depleted layer beneath the oxide scale which is formed on the surface during TMF testing, is also partly recrystallized, see [7]. The typical appearance of the deformation bands in the scanning electron microscope is as follows. What has so far been described above concerning SCA425Hf is also valid for SCA425+ but the development of a rafted fibrous structure in the deformation bands is even more pronounced due to the more severe plastic deformation in this alloy and the final fracture is more close to what is shown in Fig1.b. The rafting is also shifting between the possible slip directions creating steps and waviness in the structure as is shown in Fig. 15. It is here obvious that the rafted bands appeared first in one direction +45 to the axial direction and then -45 when the zigzag steps are created. The next step in the damage process is recrystallization but in this alloy there are also signs of fragmentation when the elongated $\gamma'$ –particles are changing into larger non uniform fragment together with a partly recrystallised $\gamma$ shown in Fig. 16.
Fig. 13 Micrographs showing crosssection SCA425Hf with deformation bands and fracture surface, (a) SEM backscatter image showing deformation band and (b) EBSD map of the same area showing only slight mis-orientation in the bands and complete recrystallization at the fracture surface.

Fig. 14 Longitudinal cross-section showing the appearance in the SEM of the bands of localized deformation in SCA425+ (backscatter image).

4. Discussion

The results from a study of damage and fracture mechanisms in three different single crystal alloys subjected to out of phase TMF-testing has been presented and will now be discussed. In earlier studies OP-TMF-damage and fracture mechanisms in single crystals have been studied [1, 7] and it was found that different mechanism like twinning was active in CMSX-4 and deformation bands with development of fibre like γ’ in SCA425Hf. This process is also active in SCA425+ tested in this study to such an extent that the final failure is ductile (Fig. 1b). What all those alloys have in common is the final part where recrystallization appears before initiation and growth of a crack. The results presented in this investigation show to a certain extent the development of the microstructure as a result of TMF-cycling but very little is known about when and why structure is changed and what influence the different microstructures have on this process.
One can at this stage only speculate about the influence of the coupling between cycling of the temperature and strain on the microstructure. One important piece of information can be found in temperature strain plots and hysteresis loops produced during testing. Aged material displayed a higher degree of inelastic strain for the midlife stress–strain hysteresis loop compared to virgin material during tests with a similar number of cycles to failure in CMSX-4. Aged material thus has a higher degree of creep relaxation during the dwell period and greater plastic deformation towards the relatively cool end of the cycle. At this stage it appears as if it is possible to modify the microstructure so that better ductility can be obtained probably at the expense of creep properties. It is also shown that one of the alloys is superior to the others but in that case probably at the expense of oxidation resistance. The exact reason why deformation bands are formed in one case and deformation twins in another is difficult to explain but important information is probably found by studying creep/relaxation response at high temperature and ductility at low temperature and what effect this has on residual stress created during cycling of strain between the two extremes and also to what extent the material show a memory effect between the cycles. A higher creep rate will probably have an effect on plasticity at low temperature and so on. The virgin CMSX-4 has a very high degree of solution hardening in the matrix and if very little shearing in combination with limited Orowan looping the twin formation is a plausible alternative while the ageing will change the microstructure by precipitation of TCP in such a way the dislocation activity is spread out and become less concentrated to even allow ductile fracture.

For the SCA425 type of alloys with smaller additions of solution strengtheners the intense rafting or elongation of γ' along the slip created during the high temperature part of the cycle will contribute to a more localized deformation at low temperature. It is
also possible to estimate the slip length from intersections to be around 5-10 μm from SEM-micrographs (Fig. 15). The formation of the lamellar bands from the general slightly axially rafted γ' structure is probably a process were γ' is rotated and elongated in the slip band under the influence of diffusion and local strain. Since there is also global rafting present in line with what is considered to be normal for γ' with negative misfit the intense rafting along the [111]-[110] slip system can be explained by [12] local strain effects[13]. This will probably allow most of the plastic deformation to take place in the matrix between the rafted bands without shearing of the γ. According to Laird et al [14] an average strain of 0.01 or more can be localized into deformation bands. Some of the γ'-particles close to the band interface are bent as if they have been dragged into the deformation band indicating a minimum of shearing of γ' taking place. This effect has been observed by Reed et al [15] and described as a rapid reorientation of γ'-raft morphology in the vicinity of localized fracture due to tri axial stress. Normally one could argue that the formation of the bands should be stopped by the axial global rafted microstructure but in this case it looks as if the γ' particles have coalesced completely and instead grown along the diagonal of the cube. There are also observations made during testing were localized processes are shown as serrated yielding on the load strain curve.

The next step in the damage process, recrystallization starting with formation of large γ'-particles in a matrix of recrystallized γ. The location of those nucleation sites is often believed to be close to intersections between bands or inclusions or other irregularities and local stress raisers. Since the deformation has become localized in the band repeated dislocation multiplication will allow nucleation and growth of new grains during the limited cycle time at a temperature below the regular higher recrystallization temperature for this alloy.

The TMF type of cycling with variations in strain and temperature has thus caused a development of microstructure influenced by variations in solid solubility within and between the coherent phase and matrix assisted by diffusion along boundaries and dislocations into a localised recrystallised structure before final rupture.

5. Conclusions

A study of the damage and fracture mechanisms of three single crystal alloys with one in two heat treatment conditions subjected to OP-TMF testing has resulted in the following conclusions.

- Heat treatment is decreasing localized damage in CMSX-4 resulting in a ductile failure
- The alloy CMSX-4 shows localized damage based on twinning
- The SCA425 type of alloy shows another type of damage based on deformation bands with rafting along the slip direction.
- Recrystallization is an active damage mechanism observed during final stages of the damage process

6. Acknowledgements

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