THERMOMECHANICAL FATIGUE OF SINGLE-CRYSTAL SUPERALLOYS: INFLUENCE OF COMPOSITION AND MICROSTRUCTURE

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Abstract

The thermomechanical fatigue (TMF) behaviour of a new high Cr-containing single crystal superalloy, known as STAL-15, is investigated. This is a candidate alloy for future industrial gas turbine (IGT) applications. TMF involves complex interactions between high and low temperature deformation mechanism, and this study highlights important factors controlling their inter-relationship. Emphasis is placed particularly on the microstructural aspects which control deformation. It is demonstrated that the TMF performance of the new alloy is comparable to that of a second generation alloy such as CMSX-4, despite the absence of Re alloying. An addition of 0.25wt% Si significantly improves the resistance to TMF further; this is attributed to a greater resistance to recrystallization and cracking along the deformation bands that develops across the cross section of the specimen during testing. The TMF resistance increases with increasing primary γ' size, but the degree of solutioning of the γ' phase caused by the solution heat treatment seems to have no significant effect.

Introduction

Nickel-based superalloys are designed to withstand extreme conditions of temperature and loading during operation. Their performance in critical hot gas path components such as blades and vanes largely limits the durability of modern gas turbines. Typically, components experience complicated and inter-related thermal and mechanical loading, especially during engine start-up and shut down. The thermal gradients in conjunction with mechanical constraints and the time variation of both the temperature and the stresses may eventually lead to thermomechanical fatigue (TMF) damage.

This paper is concerned with the TMF performance of single crystal superalloys. Broadly speaking, TMF failure in single-crystal turbine blades is promoted when plastic strains cannot be accommodated at low temperatures and creep deformation in combination with oxidation occurs at high temperature. TMF is growing in importance because gas turbines are being operated under ever more arduous conditions. The reasons for this are for example the higher degree of cyclic operation of power plants and the introduction of more advanced cooling systems which lead to steep temperature and strain gradients during operation. In combination with the strive for increasing efficiency and reduced fuel usage, the risk of TMF failure in modern gas turbines is therefore acknowledged to be increasing.

For the above reasons, there is now increased emphasis being placed on the testing of single crystal superalloys under TMF conditions. Traditionally, data from static creep testing and low cycle fatigue (LCF) tests have traditionally been used to assess the likelihood of failure; however, there is now mounting evidence to suggest that TMF is more representative of the conditions experienced during service. In this paper, attention is placed particularly on a new high Cr-containing single crystal superalloy, known as STAL-15. The effects of precipitate morphology and alloy chemistry are emphasised.

Background

Influence of TMF cycling

Constraint of free thermal expansion and contraction is an intrinsic ingredient of the thermally induced fatigue process; it is this which needs to be reproduced on the laboratory scale. By doing so, TMF testing provides a powerful tool to assess the synergistic effects of combined thermal and mechanical loads. Typically, it involves the cycling of the temperature T and mechanical strain ϵ_{mech} with different phase shifts. The most common types of TMF cycles are illustrated in Figure 1.

Figure 1: Temperature-strain cycles for different TMF tests.

The in-phase (IP) and the out-of-phase (OP) cycles represent the cases when external loads or temperature gradients are present within a component during steady-state operation. On the other hand, the clockwise-diamond (CD) and the counter-clockwise-diamond (CCD) cycles simulate the transient effects that can occur during start-up or shut-down, due to for instance different heating or cooling rates of thin versus thick sections of a component.

If one focuses on the IP-cycle, one has a situation in which the tensile stress/strain loading coincides with the high-temperature part of the cycle, see Figure 2(a). The deformation is thus characterized by high temperature creep in tension and low temperature plasticity in compression. For pure thermal loadings, this situation is found at “cold-spot areas” where the temperature is typically lower compared to the surrounding, *e.g.* near cooling holes or at structures inside cooling channels. However, the stresses/strains from thermal gradients can also be superimposed by external loads such as internal pressure and centrifugal forces which will further increase the tensile stress at elevated temperatures. The combination of high tensile stresses at an elevated temperature and significant hold times can have a very detrimental effect on fatigue performance even for rather moderate temperatures.

The situation during an OP TMF-cycle is completely different. In this case, the material undergoes creep relaxation in compression at high temperatures and plastic deformation in tension at low temperatures. A schematic illustration of the stress-strain behaviour corresponding to OP-TMF is given in Figure 2(b). This is the situation found at “hot-spot areas”, *i.e.* small regions with higher temperature than the near environment which may occur *e.g.* on a turbine blade airfoil or platform due to insufficient cooling. Since these areas will be in compression during the high-temperature running period of the engine, high-temperature creep relaxation will cause the material to be in tension at shut-down. Since rafting and other degradation mechanisms occurring at high temperature are found to have a higher effect on the low-temperature properties than on the high-temperature ones, see *e.g.* [1,2], it can be readily concluded that the OP TMF-loading condition is quite different from iso-thermal LCF-loading. The importance of the low temperature properties during TMF is also manifested by the strong influence of the minimum temperature during TMF [3,4].

In case of both IP and OP-TMF, the magnitude of the stresses in the cold end of the TMF cycle will increase with the amount of stress relaxation in the hot-end of the TMF cycle. Thus, the possibility for having reversed plasticity at engine shut-down will increase with the amount of stress relaxation during service. Thus the length of the time spent at high temperature will (in addition to other degradation mechanisms) be important for the TMF life of the component. As creep always is present when engineering components are subjected to TMF conditions, it is strongly recommended to also introduce a dwell period at the maximum temperature in the TMF-test. The length of the dwell period often varies from a couple of seconds to several hours, but for practical reasons the dwell time in laboratory tests is often much shorter than the typical time of operation for the component of interest. This is especially the case for hot components in industrial turbines where the average time between start and stop can be up to 500 hours.

Figure 2: Schematic illustration of stress versus strain during (a) In-Phase TMF and (b) Out-of-Phase TMF.

The TMF methods used for our studies are well established and accepted by both the materials and mechanics communities. In order to somewhat compensate for the shorter dwell time during laboratory testing, the TMF tests reported here were always conducted with a 20 hour dwell time in the first cycle while the following later cycles typically have a dwell time of 5 minutes.

Experimental Methods

In this paper, special focus will be placed on the newly developed superalloy STAL-15 (Ni-5Co-1Mo-3.7W-15Cr-4.55Al-8Ta-0.1Hf-0.25Si-0.03Ce), which is a new high Cr single crystal superalloy for industrial gas turbine applications [5,6]. An industrial scale investment casting facility at the University of Birmingham was used to prepare single crystal castings in the form of 15 mm diameter rods of length 150 mm. The casting stock was melted by Ross & Catherall in Sheffield, UK to industry-leading standards.

The nominal, baseline heat treatment conditions used were:

Solutioning: 1280°C for 1 h → 1300°C for 5 hrs, air cooling

Primary age: 1100°C for 6 hrs, air cooling

Secondary age: 850°C for 20 hrs, air cooling

However, as indicated below, other heat treatments as well as variations in chemical composition have been tested in order to further investigate the factors influencing the TMF behaviour of the alloy. In addition, other alloys such as SCA425 and CMSX-4 have also been studied.

Out of phase (OP) thermomechanical fatigue tests were conducted in air under mechanical strain control in the 100–950°C temperature range by the use of an MTS 810 servo-hydraulic thermomechanical 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 (T_{max}) during the first cycle. For all subsequent cycles a 5 minute hold time was applied and the total cycle time was 738 seconds. The strain ratio in all tests are $R = \epsilon_{min} / \epsilon_{max} = -\infty$.

After testing the ruptured fatigue specimens were sectioned parallel to the longitudinal axis for microstructural investigations. All samples were prepared by grinding and mechanical polishing and analysed using scanning electron microscopy. In order to obtain optimal channelling contrast in the image, an annular backscatter electron detector on a Hitachi SU70 FEGSEM operating at 10 kV was used. The contrast in such an image is associated with discontinuities in the specimen and any crystallographic defect that produces a distortion in the lattice, such as a twin, a sub-grain or dislocation can be observed. Orientation imaging microscopy (OIM) was performed using an electron back-scattering diffraction (EBSD) system from HKL Technology.

In this study the following factors have been investigated with respect to the TMF behaviour:

1. Influence of Si-addition: STAL-15 with and without an addition of 0.25% Si has been TMF tested.
2. Influence of solution heat treatment: TMF testing has been conducted on STAL-15, solution heat treated at two different temperatures, 1280°C and 1180°C, for 5 hours.
3. Influence of age heat treatment and γ' distribution: TMF testing has been conducted on STAL-15 with two different ageing conditions, 1100°C/6hours and 1120°C/24hours.

The TMF behaviour has been evaluated as the number of cycles to failure for a number of different strain ranges.

Results

Deformation and damage mechanisms

SCA425 is an example of a 1st generation (Re-free) single crystal superalloy, and its performance has been found to be significantly different from that of the Re-containing alloys on which previous studies have concentrated. Deformation is characterised by slip along the $\{111\}$ planes with shearing of the γ' -particles being more frequently observed [7]. During TMF testing, slip is concentrated into a small number of bands along the specimen gauge length and inside these bands, massive shearing of the microstructure occurs giving a very smeared and elongated appearance of the γ' -phase as illustrated in Figure 3(a). It is found that recrystallization eventually occurs along these deformation bands as illustrated by the orientation image map (OIM) in Figure 3(b) which is produced using electron back scatter diffraction (EBSD). The recrystallization is then followed by rapid crack initiation and crack growth.

Figure 3: Shearing and localized damage in SCA425 caused by OP TMF in the 100-950°C temperature range.

These observations contrast very strongly with the results of previous studies of ours and others [8,9] which have confirmed unambiguously that twinning is the main deformation mechanism in 2nd generation (Re-containing) single crystal superalloys such as CMSX-4 and TMS-82+, see Figure 4(a). Also from Figure 4(a) another common observation can be made namely that Ta, W, and Re rich μ -phase particles precipitate preferably along the twin boundaries. Sometimes it has been described in the literature that the brittle nature of the intermetallic TCP phases has a deleterious effect on the low-temperature tensile and impact strength. However we have never seen any evidence of cracking during TMF due to these precipitates. Instead, as the TMF cycling proceeds, recrystallization is found to take place within the deformation bands, with the intersection points of twins of different orientations acting as initiation sites for this process. Because of the absence of grain boundary strengthening elements, cracks can then easily initiate and propagate along the recrystallized deformation bands, see Figure 4(b).

Figure 4: Twinning and localized damage in CMSX-4 caused by OP TMF in the 100-1000°C temperature range.

Even if the main deformation mechanism during TMF of the 1st and 2nd generation single crystal superalloy seems to be different (shearing versus twinning), it is emphasised that recrystallization occurs in both types of alloys and it is this which governs the final failure of the specimens. Traditionally, recrystallization has been regarded as an issue during solutioning of as cast structures or during the rejuvenation heat treatment of in-service exposed components. However, for these situations annealing is typically performed above the γ' solvus temperature where any pinning effect due to γ' precipitates is not present, so that recrystallization can take place easily. During the above described TMF tests, the maximum temperature for which the recrystallization must have occurred at is substantially below the γ' solvus temperature.

The amount of plastic strain induced prior to the heat treatment is acknowledged to be an important factor for the recrystallization process and our results indicate that the plastic deformation introduced during OP TMF loading between 100 and 1000°C in CMSX-4 is locally so severe that recrystallization can occur even if the material exhibits the γ/γ' microstructure. The test performed on SCA425 had a slightly lower maximum temperature (950°C) compared to CMSX-4, but still all fracture surfaces exhibited significant amount of recrystallization, as seen in Figure 3(b). However, for SCA425 the damage process was found to be further enhanced by oxidation, since recrystallization occurred more easily in the γ' depleted zone under the oxide layer.

Influence of Si-addition

The addition of Si to STAL-15 has been proven to be very beneficial from an oxidation point of view [6,10]. Si promotes the formation of a continuous layer of alumina at 950°C and can thus help to bridge the critical gap between protective chromia formation at low and intermediate temperatures and protective alumina formation at high temperatures. It might be argued that the addition of Si might also potentially increase the risk of detrimental TCP phases during service at elevated temperature. However, laboratory exposures up to 10,000 hours did not reveal any significant amount of TCP phases as long as the Si content was 0.25wt% or lower [5]. A Si addition of 0.25wt% also turned out to be most beneficial from an oxidation point of view [10].

The TMF data are summarised in Figure 5. An addition of 0.25wt% Si has a strong effect on the TMF resistance of STAL-15. With the addition of Si, the TMF life increases by a factor of approximately 2.0 compared to STAL-15 without Si. The increase seems to be roughly the same for all strain ranges tested.

Figure 5: Influence of 0.25wt% Si on the TMF resistance of STAL-15.

The typical macroscopic fracture appearance of STAL-15 is illustrated in Figure 6. Several deformation bands can be seen which extend more-or-less across the complete cross-section of the specimen. Locally within these bands, pronounced shearing of the γ' structure occurs as seen in micrograph in Figure 7(a). Eventually, recrystallization occurs along these deformation bands and when this occurs cracking also takes place as illustrated by Figure 7(b).

Figure 6: Typical TMF fracture appearance of STAL-15 with 0.25wt% and solution heat treated at 1280°C.

The main deformation mechanism in STAL-15 during thermomechanical fatigue thus seems to be of the same type as for the previously investigated 1st generation single crystal alloy SCA425. When recrystallization eventually occurs along the deformation bands during the thermomechanical fatigue process, final failure is accompanied by the formation of voids due to the presence of grain boundaries and the damage process is further enhanced by oxidation ahead of the crack, since recrystallization occurs more easily in the γ' depleted zone under the oxide layer.

In specimens of STAL-15 with 0.25% Si twins were some times visible close to the fracture surface as seen in Figure 8. These twins did not extend across the complete cross section of the specimen do not seem to have a significant influence on the finale failure of the specimens. It is however interesting to notice that

this type of twinning mechanism were not seen in specimens without the Si-addition.

Figure 7: Microstructure of STAL-15 after TMF testing. (a) Deformation bands with shearing of the γ' structure. (b) Cracking along a recrystallized deformation band.

Figure 8: Twinning close to the fracture surface in STAL-15 after TMF testing.

By observing the specimens during the testing performed in the present study, it was noticed that the deformation bands on specimens with and without Si addition were developing on the surface at roughly the same number of cycles, for a given strain range. The development of deformation bands extending across the complete cross-section of the specimens was typically also accompanied by a more jerky appearance of the stress response during the test as illustrated in Figure 9. Here, the maximum stress in each stress-strain loop is plotted as a function of number of cycles in the test.

It can therefore be concluded that the addition of Si does not significantly delay the formation of deformation bands. Instead the addition of 0.25wt% Si is found to improve the resistance to developing damage such as recrystallization and cracking along the deformation bands. This improvement can at least partly be attributed to the increased oxidation resistance; however, one cannot exclude the possibility that Si also has other beneficial effects. Further investigation is needed.

Figure 9: Influence of 0.25wt% Si on the TMF resistance of STAL-15.

Influence of solution heat treatment

When CMSX-4 was tested in virgin and long term aged condition [9] it was found that the deformation mode changed significantly from very localized in the virgin material to much more dispersed in the long term aged material. The greater homogeneity of deformation promoted by ageing was associated by the extensive precipitation of TCP phases which acted as obstacles for deformation. In the long-term aged condition, none of the deformation bands were able to extend across the complete cross section of the specimen.

The interpretation of the results described above is that a more dispersed deformation behavior would be beneficial from a TMF point of view while it is reasonable to assume that a very uniform microstructure would promote localization and shorter TMF life. With this in mind, it was decided to investigate if the degree of solutioning can have a significant influence on the TMF life of STAL-15.

The Si-containing version of STAL-15 was solution heat treated at 1280°C and 1180°C respectively, for 5 hours. According to ThermoCalc [11] predictions the solvus temperature of the γ' -phase in STAL-15 with 0.25wt%Si is approximately 1240°C. Therefore as expected the higher solution heat treatment temperature resulted in a fully-solutioned microstructure. On the other hand a significant number of eutectic domains remained in the material solution heat treated at the lower temperature, see Figure 10.

Figure 10: Microstructure of material solution heat treated at 1180°C which is significantly below the γ' solvus temperature.

Figure 11 clearly illustrates that no significant influence on the TMF resistance can be expected from differences in the solution heat treatment temperature. Even the number of visible deformation bands on the specimen surface has been reduced slightly after the lower solution heat treatment temperature, compare Figure 12 with Figure 6; the fracture appearance is the same with a completely crystallographic fracture surface along one of the $\{111\}$ planes.

Thus it can be concluded that the eutectic domains are not effective obstacles for the localized deformation typically observed during TMF. This fact is also illustrated in Figure 13 where it can be seen that the deformation band can easily cut through the eutectic domains.

Figure 11: Influence of the solution heat treatment temperature on the TMF resistance of STAL-15.

Figure 12: Typical TMF fracture appearance of STAL-15 with 0.25wt% Si and solution heat treated at 1180°C

Figure 13: Deformation bands cutting through eutectic domains in STAL-15 solution heat-treated below the solvus temperature

Influence of age heat treatment and γ' distribution

During the development of STAL-15 several different primary-aging heat treatments were carried out in order to investigate the effect of differing precipitate morphologies on the mechanical properties, see reference [6]. Below the resistance to TMF is compared for two different microstructural conditions:

Microstructure 1:

First ageing at 1100°C for 6 hours

Primary γ' size is 0.39 μm

Microstructure 2:

First ageing at 1120°C for 24 hours

Primary γ' size is 0.66 μm

For both conditions the solution heat treatment was performed at 1300°C for 5 hours and the second ageing at 850°C for 24 hours. The corresponding microstructures can be seen in Figure 14.

Figure 14: Scanning electron micrographs of STAL-15 (without Si) with different first aging conditions; (a) 1100 °C for 6 h, (b) 1120 °C for 24 h

The results from the TMF testing of the two different heat treatment conditions are compared in Figure 15. Even if there are some scatter in the results, the data indicate that microstructure 2 with the largest γ' size also show the longest TMF life. The difference is approximately a factor of 1.5 in life. When these conditions were tested in creep an opposite relationship was noticed, so clearly the mechanisms controlling the TMF resistance are distinct from those determining creep. While creep resistance is determined by dislocation activity in the γ matrix phase, TMF resistance seems to be more related to the resistance to dislocation shearing of the γ' phase. As the resistance to shearing can be assumed to increase with increasing γ' size it is reasonable to also find a higher TMF resistance for microstructure 2 where the higher ageing temperature and longer ageing time results in a larger γ' size.

Figure 15: Influence of the first ageing treatment and the γ' distribution on the TMF resistance of STAL-15.

Discussion

The new STAL-15 alloy has been developed to meet the needs typically required for 1st stage turbine blades in modern highly efficient industrial gas turbine (IGT) engines. For long term robustness, this translates in turn to the need for an alloy that combines good corrosion and oxidation resistance with sufficient creep and fatigue performance. In particular, for the mechanical properties experience has shown that it is usually sufficient to have creep strength comparable to that of the best polycrystalline alloys. One can argue that the very high creep strength of alloys such as CMSX-4 is unnecessary when advanced internal cooling systems are utilized in turbine blades for IGTs, *i.e.* the internally cooled structures can carry the loads at significantly lower temperatures when good cooling systems are introduced. However, due to the thermal gradients that will be introduced an excellent TMF resistance is a necessity.

It is for this reason that the TMF behaviour is becoming of increasing importance; hence the emphasis placed on this mode of deformation in the present work. The TMF behaviour of the new alloy STAL-15 has been explored and some of the factors influencing the TMF life have been elucidated. As shown in Figure 1, creep relaxation will occur at the “hot” end of the TMF cycle. When the amount of creep relaxation is increased, the margin against plastic deformation in the cold end of the cycle will decrease, and the total amount of in-elastic strain increases for a given mechanical strain range. First of all, one can conclude that despite the lower creep resistance, STAL-15 displays almost the same level of high temperature TMF resistance as CMSX-4. This is illustrated in Figure 16. From the data, CMSX-4 has a slight advantage in high temperature TMF resistance, which in Figure 16 can be seen to be about 50°C temperature advantage. This can partly be attributed to the better creep resistance as argued above but it is also likely that the twinning mechanism seen in the 2nd generation of single crystal alloys is beneficial from a TMF point of view. The reason is that the degree of severe localization, as seen in for instance Figure 6, decreases with the twinning mechanism.

However, in the real situations which arise in the turbine, one needs to point out that the effects are somewhat more complicated. The potentially negative influence of the localization may not be dramatic since it will be difficult in practice for deformation bands to develop over the same distances as in these laboratory tests on smooth specimens. The reason for this is that any temperature and strain gradient will imply constraints over the distance the bands can develop, particularly in the thicker sections such as turbine blade platforms which are the prone to TMF damage. But further research is needed to elucidate the influence of TMF on the mechanical degradation mechanisms which are pertinent to these components.

Figure 16: Comparison of TMF data for STAL-15 and CMSX-4. Data for CMSX-4 are taken from [9,12]. Note that no direct comparison at the same temperature range is possible, but otherwise the test conditions are equivalent (dwell times, temperature rates, R-ratio's etc)

Summary and Conclusions

The thermomechanical fatigue (TMF) performance of a new Cr-containing single crystal superalloy known as STAL-15 has been investigated. It is a candidate for use in future designs of industrial gas turbines (IGTs) for power generation applications. Emphasis has been placed particularly on the microstructural aspects which determine resistance to TMF. Our specific conclusions are:

1. The TMF performance STAL-15 has been shown to be comparable to CMSX-4, despite the absence of Re alloying. This confers considerable benefits in terms of cost.
2. An addition of 0.25wt% Si significantly improves the resistance to TMF displayed by STAL-15. This improvement is attributed to a greater resistance against recrystallization and cracking along the deformation bands that develops across the complete cross-section of the specimen.

3. The degree of solutioning of the γ' phase at the solution heat treatment has been found to have no significant effect on the TMF resistance.
4. TMF resistance seems to increase with increasing γ' size. This can be attributed to higher resistance to shearing of the γ' particles as their radius increases.
5. The main deformation mechanism are different in STAL-15 and CMSX-4. While the Re containing second generation single crystals exhibits twinning as the main deformation mechanism, STAL-15 and other first generation single crystals mainly display a shearing of the γ' .

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