Effect of rejuvenation heat treatment on microstructure and hot corrosion resistance of a service-exposed nickel-based gas turbine blade

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Abstract
The present work was carried out to evaluate the effect of rejuvenation heat treatment on recovery of the microstructure and hot corrosion resistance of a service-exposed nickel-based gas turbine blade. Different rejuvenation heat treatment cycles were carried out on the service-exposed blade. The microstructure of the blades was examined by scanning electron microscope (SEM) in as-received condition and after rejuvenation heat treatment. Both of the service-exposed and rejuvenated blades were subjected to cyclic hot corrosion tests to evaluate the resistance to hot corrosion. Microstructural investigations showed that the rejuvenation heat treatment cycle, including solution treatment at 1175 °C for 3h, followed by air cooling and two-stage aging treatments at 925 °C/1h and 845 °C/24 h was most successful to recover the microstructure of the service-exposed blade to its virgin condition. Moreover, it was found that the rejuvenation heat treatment has a significant effect on the hot corrosion resistance of the service-exposed blade via dissolution of continuous grain boundary carbide films and the redistribution of the elements in the alloy matrix. The results of the hot corrosion experiments showed that the rejuvenation heat treatment can improve the hot corrosion resistance of the service exposed blade up to about 60 percent.

1. Introduction
Turbine blades made of nickel-based superalloys are one of the most important hot section components in gas turbine systems. They are subjected to complex combinations of inhomogeneous stresses and temperature distribution as well as high-temperature corrosion and oxidation environment [1]. The most important service induced microstructural changes of the nickel-based superalloys are consisting of coarsening and coalescence of the gamma prime particles, formation of continuous carbide precipitates on the grain boundaries and topologically close-packed (TCP) phase formation [2–4]. These changes in microstructure bring about degradation of the mechanical properties such as tensile strength, fracture toughness, and creep resistance of the alloy [5–8]. Resistance to hot corrosion is one of the other important properties of the nickel-based superalloys, which can be affected by the microstructural changes resulted from high-temperature operation condition of the gas turbine blades [9]. Hot corrosion is one of the main reasons for blade failure in the hot section of the gas turbines. This phenomenon consumes the material and consequently, reduces the load-carrying ability of the components [10]. Formation of corrosive molten alkali salt contaminants such as Na2SO4, NaCl, and vanadium compounds such as V2O5 on the surface of the turbine blades can result in the protective oxide layer on the blades to be damaged and leads to accelerated corrosion named hot corrosion [11].

The high cost of the turbine blades has led to an increased industrial interest in extending their operation lives by various repair and rejuvenation procedures. The rejuvenation heat treatment processes can provide a way to restore the properties and microstructure of the long-term used gas turbine blades to their original condition [12, 13]. This heat treatment consists of two main stages of solution and aging treatments. A primary
solution treatment to dissolve as much of the microstructural phases to achieve a single phase microstructure and then an aging treatment to form a desired microstructure. The rejuvenation heat-treatment for precipitation strengthened nickel-based superalloys is a relatively complicated process. Based on the literature, repeating the alloy’s standard heat treatment sequence does not always work to fully recover the microstructure and reestablish the properties of the aged alloys. This makes the selection of heat treatment parameters increasingly challenging for precipitation strengthened nickel-based superalloys.

Many reports have been published on the rejuvenation of nickel-based superalloys in the past few years. Most of these studies referred to the effect of the rejuvenation treatment on the mechanical properties of the alloys, but there is only a small amount of investigation involved the relationship between rejuvenation treatment and hot corrosion resistance of the nickel-based superalloys. Thus, from the point of view of application, it is necessary to evaluate the effect of rejuvenation treatment on hot corrosion resistance of the service exposed nickel-based gas turbine blades.

This work aims to investigate the effect of rejuvenation heat treatment on microstructural recovery and hot corrosion resistance of a 100,000 h service-exposed nickel-based gas turbine blade. For this purpose, a service-exposed blade made of Inconel 738 alloy was subjected to different cycles of rejuvenation heat treatment. Afterward, hot corrosion experiments were performed on the service-exposed and rejuvenated blades. Microstructural characterization using SEM, EDS, and XRD was carried out on hot corroded samples to investigate the changes in hot corrosion mechanisms before and after the rejuvenation heat treatment.

Table 1. Chemical composition of the Inconel 738 gas turbine blade (wt%).

| C  | Co  | Cr  | Mo  | W  | Ta  | Al  | Ti  | Fe  | Ni  |
|----|-----|-----|-----|----|-----|-----|-----|-----|-----|
| 0.17 | 8.98 | 15.82 | 1.57 | 2.48 | 1.54 | 3.26 | 3.30 | 0.23 | Bal. |

Table 2. Different heat treatment programs applied for recovering the blade microstructure.

| Program no. | Solution (air cooling) | Aging-step 1 (air cooling) | Aging-step 2 (air cooling) |
|-------------|------------------------|-----------------------------|-----------------------------|
| 1a          | 1120 °C/2 h            | —                           | 845 °C/24 h                 |
| 2           | 1175 °C/3 h            | 925 °C/1 h                  | 845 °C/24 h                 |
| 3           | 1175 °C/3 h            | 1055 °C/1 h                 | 845 °C/24 h                 |
| 4           | 1200 °C/3 h            | 925 °C/1 h                  | 845 °C/24 h                 |
| 5           | 1200 °C/3 h            | 1055 °C/1 h                 | 845 °C/24 h                 |

* Standard heat treatment (SHT).

Figure 1. (a) The service-exposed gas turbine blade and (b) The contour of temperature distribution on the blade geometry.
Figure 2. (a) and (b) SEM micrographs of γ′ precipitates and grain boundary carbides at root of the service-exposed blade, respectively, (c) and (d) SEM micrographs of γ′ precipitates and grain boundary carbides at top of the airfoil of the service-exposed blade, respectively (e) and (f) SEM micrographs of γ′ precipitates and grain boundary carbides at top of the airfoil of the blade after rejuvenation with optimum heat treatment cycle, respectively, (g) EDS analysis result of the accumulated grain boundary carbides (point A in figure 2(d)).
2. Experimental procedure

First stage blades of a 3 megawatt (MW) ground gas turbine with 100,000 h service-exposure were used in this study. The blades were made of Inconel 738 nickel-based superalloy with the chemical composition shown in table 1.

Temperature distribution on the blade was determined by numerical analysis using computational fluid dynamics (CFD) method to obtain the zones with the minimum and maximum temperatures. To evaluate the effect of high-temperature operation condition of the gas turbine on the alloy microstructure, samples were cut from the zones shown in figure 1(a). For metallographic investigations, the samples were ground, polished, and etched by using 1%HF + 33%HNO3 + 33%CH3COOH + 33%H2O solution. Microstructural investigations performed on the service-exposed blade using SEM. An overview of the gas turbine blade and the locations of metallographic observations are shown in figure 1(a). The contour of temperature distribution on the blade geometry is also presented in figure 1(b).

At the next step, samples from the zone with the maximum temperature (top of the airfoil at the leading edge of the blade) were cut and subjected to the rejuvenation heat treatments. Five different heat treatment programs were used to recover the blade microstructure, as listed in table 2. The microstructure of the rejuvenated specimens after various heat treatment programs were characterized by SEM.

Afterward, specimens with dimensions of 10 × 10 × 1 mm from the service-exposed and rejuvenated blades were cut and subjected to hot corrosion tests. The rejuvenation program that performed before the hot corrosion tests was the optimum condition achieved in the first stage experiments, i.e., 1175 °C/3 h + 925 °C/1 h + 845 °C/24 h.

For hot corrosion tests, the specimen surfaces were prepared by grinding on SiC papers up to 1200 grit size and ultrasonically cleaned in an acetone bath. The specimens were sprayed with a saturated solution of 75 wt% Na2SO4 + 25% K2SO4 in distilled water and then dried on a heating plate. The amount of deposition was controlled by weighting the specimens before and after salt spraying to achieve a salt supply of about 2 mg cm⁻² on the surface. Hot corrosion tests were performed in an electrical resistance tube furnace at 900 °C for 120 h. To measure the corrosion kinetic, the tests were interrupted every 24 h and the changes in weight of the specimens were recorded by an AND-FX200 electronic balance with an accuracy of 1 mg. After hot corrosion tests, the cross-section of the corroded specimens was examined using a VEGA3 XMU-TESCAN SEM equipped with energy dispersive spectrometer (EDS). The phase constituent of the corroded specimens was identified using a PW 1840 PHILIPS x-ray diffractometer with Cu Kα radiation, λ = 1.54056 Å, and diffraction angle range =10–90°. The interpretation of the x-ray diffraction (XRD) results was carried out by PANalytical X’Pert High Score software.

3. Results and discussion

3.1. Rejuvenation

Figure 2, illustrates typical microstructures from the root zone of the service-exposed blade, and also typical microstructures from top of the airfoil of the service-exposed blade, before and after the rejuvenation heat
treatment. Figures 2(a) and (b) show SEM micrographs of γ’ precipitates and grain boundary carbides at root of the service-exposed blade, respectively. As shown in figure 2(a), ultra-fine γ’ phases with cubical morphology are dispersed in the matrix. On the other hand, according to the micrograph shown in figure 2(b), discontinuous carbides are visible at the grain boundaries. According to figure 1(b) and also based on the previous investigations, since the root of a turbine blade is located farthest from the hot gas path, the service temperature of the root zone is relatively low and did not cause to the degradation of the microstructure [12, 15]. Therefore, it can be examined and deemed to be representative of the pristine/as-manufactured microstructure and the comparison of the microstructures of root and other zones of the blade can be used to determine the changes generated by the service.

SEM micrographs shown in figures 2(c) and (d) are related to top of the airfoil of the service-exposed blade at the leading edge. It is clear that the airfoil microstructure at top of the leading edge has aged significantly compared to the pristine condition of the root microstructure due to exposure to higher service temperature.

This is consistent with the results of the numerical heat transfer analysis presented in figure 1(b). As shown in figure 2(c), the γ’ precipitates have coarsened and begun to coalesce, and their original cubical shape has changed into the spherical platelets. Furthermore, needle-like TCP σ phases have been formed and are visible in the microstructure.

Figure 4. SEM micrographs from the cross-section of the hot coddled specimens (a) top of the blade before rejuvenation (b) top of the blade after rejuvenation (c) root of the blade before rejuvenation and (d) root of the blade after rejuvenation.

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On the other hand, based on the micrograph shown in figure 2(d), the accumulation of carbides at grain boundaries is the other significant phenomenon that occurred as a result of long-time exposure to high-temperature service. According to the result of EDS analysis shown in figure 2(g), the carbides accumulated at grain boundaries (point A in figure 2(d)) are mainly composed of $M_23C_6$ ($M$ is Cr, Mo, W) and $M_6C$ ($M$ is Mo, W) carbides.

All of these deleterious microstructural changes, i.e., $\gamma'$ coarsening, formation of TCP $\sigma$ phase and accumulation of carbides at grain boundaries can reduce the mechanical properties and toughness of the alloy and facilitate the crack initiation and propagation [12, 16, 17].

The microstructures of the rejuvenated blade at top of the airfoil are presented in figures 2(e) and (f). Comparing the micrographs shown in figures 2(e) and (f) with the micrographs illustrated in figures 2(a) and (b), it can be seen that the rejuvenated microstructures of the service-exposed blade are very similar to the microstructures of the root zone of the blade which almost can be considered as the original microstructure of the alloy. As shown in figure 2(e), after rejuvenation heat treatment, fine cubical $\gamma'$ phases with an average size of about 0.4 $\mu$m have been formed and dispersed in the matrix. Needle-like TCP $\sigma$ phases have also dissolved and disappeared entirely after the rejuvenation heat treatment.

Furthermore, as shown in figure 2(f), the rejuvenation heat treatment has largely led to the replacement of continuous grain boundary $M_23C_6$ and $M_6C$ carbides with the discrete precipitations. Accordingly, it can be concluded that the applied rejuvenation heat treatment program almost has restored the damaged microstructure of the service-exposed blade.

### 3.2. Hot corrosion

#### 3.2.1. Weight change and corrosion kinetic

Figure 3 represents the relative weight change (the weight change/surface) versus time of the specimens during exposure to hot corrosion. It is observed that for all specimens (except top of the service-exposed blade before rejuvenation) the relative weight gain is increased up to 24 h and subsequently shows weight loss to 72 h. With further exposure time to 120 h, the relative weight changes are decreased and have a slight fluctuation. The decrease or increase in the relative weight change during exposure to hot corrosion is related to the formation of oxide scales or their delaminating in continue.

During the first stage (up to 24 h), the transportation of chromium ions to the surface and formation of protective oxide led to an increase in relative weight change. In continue, up to 72 h, the decrease in relative weight change can be attributed to the damaging of the protective layer by thermally induced stresses. It can lead to scale spallation from the base metal surface. In this condition, the corrosion rate is controlled by accelerated diffusing of chromium ions to surface and oxygen atoms to oxide–metal boundary via damaged path of the oxide layer. In general, negative mass change is due to the exfoliation of solid reaction products. The slight
increase in weight change with the exposure time is probably due to the growth of the non-protective spinel oxide layer [18–21].

For the specimen from top of the service-exposed blade before rejuvenation, the decrease in weight change in the initial stage of the hot corrosion test and its significant negative mass gain up to the final of the test is associated to occurring of rapid hot corrosion attack and formation of a non-protective oxide layer.

3.2.2. Microstructural characterization of hot corroded samples
The microstructure of the cross-section of the service exposed and rejuvenated specimens after cyclic hot corrosion tests are presented in figure 4.

Figures 4(a) and (b) are related to the samples from top of the blade and figures 4(c) and (d) are related to the samples from root of the blade. Formation of surface oxide layers and the occurrence of internal oxidation are visible obviously in these figures. As can be seen, the rejuvenation heat treatment has resulted in a significant

Figure 6. EDS analysis results from different points in figure 5, (a) point marked A, (b) point marked B, (c) point marked C.
difference in hot corrosion behavior of the samples from top of the blade. In the service exposed and non-
rejuvenated sample, the corrosion products penetrated significantly through the boundary and surface
degradation has been occurred significantly (more than 200 microns) in comparison to the rejuvenated sample.

The formation of thick M$_2$C$_6$ carbides layers through the grain boundaries has created a continuous and
easy path for penetration of corrosive molten salts and oxygen in the non-rejuvenated sample which can result in
severe internal destruction. Furthermore, the accumulation of chromium in grain boundaries and aluminum in
γ' deposits has eliminated the protective role of these elements. On the other hand, as shown in figure 4(b), after
rejuvenation and modification of the microstructure, the depth of the oxygen penetration has significantly
reduced which resulted in better hot corrosion resistance. It seems that the undesirable microstructural
conditions in top of the blade, in the non-rejuvenated sample have caused severe damage during the hot
corrosion tests.

According to the micrographs shown in figures 4(c) and (d), the samples from root of the blade have been
attacked uniformly and the oxide layer is present on their surfaces. It is clear that the oxygen and corrosive salts
are introduced through the boundaries (cracks of oxide layer), but their penetration depth is low (about 30
microns). As a result of the lower temperature of root area and the lack of microstructure changes during service,
the microstructure is almost the same before and after the rejuvenation treatment, and so no significant
difference can be considered in the microstructure of the corroded samples from the root of the blade before and
after the rejuvenation heat treatment.

Figure 5 shows a higher magnification SEM micrograph from the cross-section of the hot corroded sample
from top of the blade before rejuvenation treatment. The corresponding EDS point analysis results of this
sample are shown in figure 6. According to the results of EDS at point A (figure 6(a)), the surface oxide layer
contains various elements such as Al, Ti, Ni, and Cr. In addition, it is clear that the chromium content is lower
than other elements which can be due to the accumulation of Cr at the grain boundaries as carbide precipitates.

By placing the molten salt on the surface of the alloy, the protective oxide layer is attacked significantly due to
the corrosive behavior of the molten salt. It is clear that the area under the oxide layer is emptied from some of
the elements and a porous layer is formed. It is believed that the protective oxide layer is dissolved in the molten
salt, and the oxygen and sulfur ions penetrated through the porous layer and into the alloy. These ions react with
the surface of alloys and form sulfide and oxide compounds inside the alloy. Inter-granular corrosion and sulfide
particles are indicative of Type I of hot corrosion [22, 23].

Figure 7 shows a higher magnification SEM micrograph from the cross-section of the hot corroded sample
from top of the blade after rejuvenation treatment. The corresponding EDS point analysis results of this sample
are shown in figure 8. The protective oxide layer in this sample contains various elements such as Al, Ti, Ni, and
Cr. As can be seen the surface oxide layer has a high amount of chromium content (figure 8(a)). Proper
distribution of chromium after the rejuvenation treatment has allowed the formation of a protective layer on the
surface of the alloy. At point A, the amount of Al on the surface is low. Al requires less activity of O for oxidation.
Figure 8. EDS analysis results from different points in figure 7, (a) point marked A, (b) point marked B, (c) point marked C, (d) point marked D.
than Cr and Ni. Thus it is expected that Al has oxidized at depth of sample as dispersed oxides. The chemical composition analysis of point D (figure 8(d)) confirms this issue.

3.2.3. XRD analysis of hot corroded samples
Figure 9 shows the results of XRD test of the service exposed and rejuvenated specimens after cyclic hot corrosion. Figures 9(a) and (b) are related to the samples from top of the blade and figures 4(c) and (d) are related to the samples from root of the blade. As can be seen, the oxide layers formed on the surface of the samples after hot corrosion are composed of chromium, titanium, nickel and aluminum oxides. In addition, some other components such as \( \text{Ti}_2(\text{SO}_4)_3 \), NiS and CrS have formed on the samples due to presence of the sulfur element.

According to the XRD patterns shown in figure 9, the oxide layers formed on the surface of the hot corroded specimens are not same for the service exposed and rejuvenated samples. The oxides on the surface of the service exposed sample are mainly composed of aluminum and titanium oxides. That is while for the rejuvenated sample chromium oxide also has formed on the surface during the hot corrosion test.

During hot corrosion, by crusting/spalling the initial \( \text{Cr}_2\text{O}_3 \) layer, due to the affinity of Cr to reaction with oxygen, the subsequent \( \text{Cr}_2\text{O}_3 \) is formed. This leads to depletion of Cr in the surface of alloy and so in continue, other oxide phases from Al, Ti, and Ni elements will be created. However, in the service exposed and non-rejuvenated sample, since Cr is engaged in chromium carbide compounds at grain boundaries, no protective \( \text{Cr}_2\text{O}_3 \) layer can be formed at the initial state of the hot corrosion \cite{24–26}. This results in higher negative weight changes for this sample (figure 3) and then lower hot corrosion resistance.

4. Conclusions

(1) The rejuvenation heat treatment cycle, including solution treatment at 1175 °C/3 h A\(^{-1}\)C\(^{-1}\) (air cooling) followed by two-stage aging treatments at 925 °C/1h and 845 °C/24 h was the most successful cycle to recover the microstructure of the service-exposed blade to its virgin condition.

(2) After the rejuvenation heat treatment, ultra-fine \( \gamma' \) phases with cubical morphology were dispersed in the matrix, discontinuous carbides formed at the grain boundaries, and needle-like TCP \( \sigma \) phases were also dissolved and disappeared from the blade alloy microstructure.
(3) The rejuvenation heat treatment has a significant effect on improving the hot corrosion resistance of the service-exposed blade via the dissolution of continuous grain boundary carbide films and the redistribution of the elements in the alloy matrix. This heat treatment can improve the hot corrosion resistance of the service exposed blade up to about 60 percent.

(4) In the service exposed samples since Cr is engaged in the chromium carbide compounds at grain boundaries, protective Cr₂O₃ oxide layer cannot be formed at the initial state of the hot corrosion, which results in higher negative weight change for non-rejuvenated samples and then lower hot corrosion resistance.

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