Study of the structure and chemical composition of the protective coating of a fist stage gas turbine blade after regenerative heat treatment

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Abstract. Investigation of the structure and chemical composition of the protective coating of the first stage IN738 gas turbine blade after standard regenerative heat treatment was done. It was found the degradation of microstructure and chemical composition of both the blade feather and its protective coating. Redistribution of the chemical elements decreasing the corrosion resistance was observed inside the protective coating. Cracks on the boundary between the blade feather and the protective coating were found by scanning electron microscopy. The carbide transformation and sigma phase were found in the structure of the blade feather. Based upon the structural and chemical composition studies, it is concluded that the standard regenerative heat treatment of the IN738 operative gas turbine blade does not provide full structure regeneration.

1. Introduction
Heat-resistant nickel alloys are well-known materials for the manufacture of gas turbine blades operating at high temperatures and aggressive environments. Gas turbine blades made from heat-resistant nickel superalloys work in the aggressive corrosive gas environment; hot corrosion is one of the main problems of these materials. Special alloying and application of protective coatings are usually used to improve the corrosion properties of these materials.

The Inconel 738C (IN738) super alloy, developed in 1968, is one of the important nickel based super alloys. This alloy shows the improved creep, hot corrosion, and oxidation resistance and is used in land-based gas turbines [1]. The working temperature of the first stage INC738 gas turbine blade is about of 1100 °C [2]. The IN738 super alloy contains refractory elements such as Mo, W, Ta, Cr and Co to prevent local hot corrosion. This super alloy resists oxidation to 1093 °C and shows the improved sulfidation resistance up to 982 °C [1].

In addition to alloying of refractory elements, the different corrosion resistant coatings are used for nickel gas turbine blades. Aluminum coatings have the ability to form a protective oxide film of Al2O3 allowing the material to work at temperatures above 900 °C [3]. Yttrium can be added into the aluminide coating for increasing the resistance of the INC738 gas turbine blade against oxidation and hot corrosion [4]. The most effective hot corrosion resistance alloy element is chromium. The presence of chromium in the alloy reduces the amount of aluminum needed for the formation of a protective Al2O3 oxide [5]. The chromium combines with oxygen to form Cr2O3, which prevents dissolution of protective oxide [2]. MCrAlY coating in which M = Ni, Co or Fe, is successfully used for IN738 superalloy to increase the corrosion protection [5-6]. Co-36.5Ni-17.5Cr-8Al-0.5Y protective coating is
applied by low pressure plasma spraying [6] or electrolytic deposition on hot sections of gas turbine [5]. Both processes are followed by a homogenization heat treatment at 1040-1120 °C [5].

During the exploitation gas turbine blade is in stressed thermo-mechanical conditions. Degradation process occurs in both the internal structure of the blade and protective coating of the blade. Structure recovery under heat treatment is important process determining service lifetime of the gas turbine blade.

In this article we present the investigation of the structure and the chemical composition of the protective coating of the operating IN738 gas turbine blade after standard regenerative heat treatments.

2. Experimental

Standard regimes of the regenerative annealing were described in [7]. Service-exposed IN738 blade of the first stage of a gas turbine after a standard two steps regenerative annealing (1121 °C for 2 hr, air cool then 843 °C for 24 hr, air cool) was used for investigation. The annealing of the blade after standard exploitation time was done in accordance to the service plan. The study was carried out using an optical microscope Micromed MET and a scanning electron microscope JSM 6490 with the energy dispersive and wave micro-analyzer Oxford Inca. Small pieces were cut from the different parts of the blade feather for study. The MCrAIY coating was applied by vacuum plasma spray process.

3. Results and discussion

The inner part of the blade feather, which is bordering with the cooling system, is presented in Figure 1a. Thickness of the protective coating in this part of the blade is about of 50 μm. The thickness of the protective coating in the outer part of the blade feather is larger than that in inner part (Figure 1b). This part of the blade works at hot temperature and is more stressed.

Structure of the blade feather is shown in Figure 2. Carbides are observed inside the grains and their boundaries. Intragranular carbides have a well-defined globular shape which is typical for primary MC carbide. Two types of the carbides are observed at the grain boundaries: the primary MC carbide with the globular shape and secondary M23C6 carbide with an elongated shape (Figure 2a). It is known that M23C6 carbides in nickel super alloys are mainly deposited from the matrix during heat treatment and service (at 760–980 °C) [8]. M23C6 carbide may consist of chemical elements such as Cr, W and Mo. This type of carbide is formed due to super-saturation of carbon in the matrix and degeneration of the MC type carbide [8]. The precipitations of the irregular shape distributed along the grain boundaries and inside grains are associated with the σ-phase (Figure 2b). This phase is enriched with nickel, tantalum, titanium, niobium, and molybdenum.

Needle-shaped precipitates, presumably borides, were found at the boundary between the coating and the outer part of the blade feather (Figure 3a). The straight determination of the chemical composition of these precipitates is difficult due to the limitations associated with the size of the electron
microscope probe. Attention should be paid that the morphology of the hardening intermetallic $\gamma'$-phase near the boundary with the protective coating is changed (Figure 3b). Intermetallic $\gamma'$-phase precipitates have elongated shape and are bigger in size than the $\gamma'$ cuboids of the blade feather (Figure 3b).

![Image](attachment:image_url)

**Figure 2.** Structure of the blade feather, SEM.

![Image](attachment:image_url)

**Figure 3.** Microstructure of the $\gamma'$-phase near the protective coating of the outer part of the blade feather.

The EDS analysis supports these observations. Secondary carbide $\text{M}_2\text{C}_6$ has a high content of nickel, chromium, and cobalt. EDS analysis of the primary MC carbide shows a high content of titanium, niobium, and tantalum; chromium is practical absent. According to the literature data, hardening of the grain boundaries in heat-resistant nickel superalloys is achieved by MC type carbides based on Nb, Ti, W. To ensure a high heat resistance, carbides should have the globular shape, size of about 1 $\mu$m or less. MC carbides should be uniformly distributed along the grain boundaries without formation of a continuous grid. The probability of the formation of topologically close-packed (TCP) phases ($\sigma$, $\mu$, Laves phases), as well as of $\text{M}_6\text{C}$ or $\text{M}_2\text{C}_6$ carbides, leading to softening of the alloy, should be minimized [7]. The type of carbide transformations and TCP phases depends on alloy doping and operating temperature. According to the literature, carbides $\text{M}_2\text{C}_6$ and $\sigma$-phase may form in the IN738 super alloy after the exploitation for a long time at high temperature [1, 9]. The appearance of $\sigma$-phase as well as $\text{M}_2\text{C}_6$ carbides points to the degradation of the alloy composition and leads to embrittlement.

Figure 4 presents the microstructure of the protective coating of the inner part of the blade feather. The temperature and stress distributions in the different parts of the blade feather were presented in [10]. According to [10], the inner part of the blade feather is also at high temperature but the bending stress in this part is lower than that in outer part.
In our blade the protective coating of the inner part of the blade feather was applied on the edge zone near the cooling system. According to EDS analysis, the chemical composition of this coating is complicated by the diffusion of the elements from the blade feather (Table 1).

Chemical composition of the protective coating of the outer part of blade feather has a high percentage of oxygen, aluminum, nickel, chromium, and yttrium. Numerous longitudinal cracks running along the coating are found by scanning electron microscopy (Figure 5b). The oxygen and aluminum content in coating of this part of the blade feather is much lower than that in the inner part. Yttrium, chromium, cobalt and nickel are also present in the composition of the coating. Redistribution of the concentrations of the chemical elements inside the protective coating is observed (Table 1). The EDS results of the chemical composition of the blade feather are given in Table 2.

![Figure 4](image)

**Figure 4.** Structure of the protective coating in the inner part of the feather and EDS spectrum taken from the Area 2.

![Figure 5](image)

**Figure 5.** Structure of the protective coating in the outer part of the blade feather.

**Table 1.** Chemical composition of the protective coating in the different areas, EDS analysis from marked areas in Figures 4-5.

| Area | Ni  | Al  | Co  | Cr  | Y   | Si  | Ta  | O   | C   | Fe  | Zr  | Nb  | Mo  | Ti  |
|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 2    | 29.93 | 18.66 | 14.03 | 14.03 | 1.03 | 0.74 | 2.67 | 6.69 | 5.96 | 0.66 | -   | -   | 1.48 | 4.12 |
| 3    | 29.55 | 2.01  | 34.07 | 24.65 | 1.08 | 0.43 | 1.38 | 1.36 | 2.45 | 0.46 | 0.34 | 0.41 | 1.08 | 0.73 |
| 4    | 53.58 | 2.51  | 9.07  | 14.95 | 1.14 | 1.68 | 3.23 | 0.79 | 0.59 | 0.69 | 1.09 | 1.88 | 5.21 | 3.59 |
Table 2. Chemical composition of Inconel 738C, wt.% (marked Area 1 in Figure 2b).

|   | Ni   | Cr   | Co   | Mo   | Ta   | Al   | Ti   | C    | Zr   | Fe   | Nb   |
|---|------|------|------|------|------|------|------|------|------|------|------|
|   | balance | 15.23 | 8.73 | 3.84 | 5.02 | 2.18 | 3.64 | 1.4  | 0.89 | 0.68 | 1.19 |

It can be seen from the Tables 1-2 that the degradation of coating occurs not only due to oxidation. The area of the protective coating, which is close to the boundary with the feather (Area 4 in Figure 5b), has the chemical composition which is close to that of the blade feather (Area 1 in Figure 2). In this case, we can say about the diffusion redistribution of the chemical elements between blade feather and protective coating. The process of redistribution of the chemical elements may be explained by the Gorsky effect which was observed previously in the gas turbine blade after long service time in [11]. This effect deals with ascending diffusion under elastic-plastic conditions. Under stress, the atoms with small diameter move into the compressed regions and atoms with bigger diameters move into the stretched regions. It is known that the different parts of the working gas turbine blade are under different stress-temperature conditions [10]. The upper edge of the blade feather is subjected to the greatest stresses and temperatures in comparison with that of the inner part of the blade feather which is bordering with the cooling system. In this study we investigate the upper edge of the blade feather and part near the cooling system. Comparison between Table 1 and 2 shows that the oxygen concentration in the coating of the inner part of the blade feather is increased as high as 6.69% and cobalt and chromium contents are reduced to 14.03%. The coating of the outer part of the blade feather has less oxygen content but aluminum content is significantly reduced. Such decrease in content of aluminum testifies that coating does not protect the base metal.

4. Conclusion

The following conclusions can be done from this study:

1. The cracks on the boundary between the feather blade and protective coating are observed. This fact indicates a low adhesion and the possibility of peeling off the coating. Redistribution of the chemical elements decreasing the corrosion resistance is observed in the protective coating.

2. It is found the degradation of microstructure and chemical composition of both blade feather and protective coating. The degradation process is caused by stress, high temperature, hot corrosion, and oxidation. Service induced MC to M₂₃C₆ carbide transformation and formation of TCP σ-phase indicate the redistribution of the chemical elements inside the material of the blade feather.

3. Based upon the structural and chemical composition studies, it is concluded that the standard regenerative heat treatment of the IN738 operative gas turbine blade does not effect on carbides transformation, TCP σ-phase dissolution, and the protective coating, and thus do not guarantee the full recovery of the IN738 gas turbine blade.

Acknowledgments

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