Analysis of phase transformations in Inconel 738C alloy after regenerative heat treatment

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Abstract. Study is based on the characterization of the chemical composition the phase transformations in Inconel 738C gas turbine blade after standard regenerative heat treatment. The microstructure and chemical composition were examined by scanning electron microscope and transmission electron microscope equipped with an energy dispersive X-ray spectrometer. It was found the degradation of microstructure of the blade feather. Redistribution of the chemical elements decreasing the corrosion resistance was observed inside the blade feather. The carbide transformation and sigma phase were found in the structure of the blade feather. It is found that the standard regenerative heat treatment of the IN738 operative gas turbine blade does not effect on carbides transformation, TCP $\sigma$-phase dissolution, and thus do not guarantee the full recovery of the IN738 gas turbine blade.

1. Introduction

Heat-resistant nickel alloys are composite structural materials. The complex chemical composition of these alloys is necessary for the formation of the structure, phase composition, and stability of the alloy under long-time exposure. Intermetallic $\gamma'$-phase based on Ni$_3$Al ($\gamma'$-phase, L1$_2$-type superstructure) and solid solution ($\gamma$-phase, FCC) are the main phases of the nickel superalloy. Cobalt, chromium, molybdenum, and tungsten are the strengthening elements of the nickel solid solution. In addition, chromium plays an active role in the protection of the alloys against oxidation. The volume fraction of the strengthening $\gamma'$-phase in the nickel super alloys depends on the aluminum content. Increase in the $\gamma'$-phase volume fraction at the operating temperature improves the long-term strength of the alloy [1].

In the carbon-containing nickel superalloy, MC-type carbides are formed – for example, NbC or TiC. During blade operation (with heating), the carbide reactions with the formation of the secondary M$_{23}$C$_6$-type carbides take place. Primary MC-type carbides and $\gamma'$-phase promote these reactions [2]. An important aspect of phase stability is the stability of the superalloy against the formation of excess intermetallic phases. The topologically close-packed phases (TCP-phases), such as the $\sigma$-phase, $\mu$-phase or Laves phases are brittle and have unfavorable lamellar morphology [3].

The heat resistance of the nickel super alloy is determined by the complex interactions of several factors, such as:

- Growth dispersion of the crystalline structure and its perfection (small misorientations between the grain blocks);
- Volume fraction of the strengthening intermetallic $\gamma'$-phase, its morphology and dispersion distribution;
- Lattice mismatch between the $\gamma$-solid solution and intermetallic $\gamma'$-phase;
Content of the intermetallic TCP-phases;
- Volume fraction and morphology of the carbide particles [4].

For heat resistance, the most important factor is the thermal stability of the strengthening $\gamma'$-phase. The diffusion processes take part in the phase formation, coagulation, and dissolution. In conditions of high stresses, the effect of diffusion becomes especially important [5].

The Inconel 738C (IN738) super alloy, developed in 1968, is one of the important nickel-based super alloys. This alloy shows improved creep, hot corrosion, and oxidation resistance and is used in land-based gas turbines [6, 7]. The working temperature of the first stage INC738 gas turbine blade is about 1100°C [8]. The exploitation of the gas turbine blade occurs under stressed thermo-mechanical conditions. Degradation process including phase transformations occurs in structure of the blade under operation. Structure recovery under heat treatment is important in determining service lifetime of the gas turbine blade.

The main objective of this research is to study the degradation phenomena occurring in strengthened Inconel 738C alloy subjected to standard regenerative heat treatment.

2. Methodology
Standard regimes of the regenerative annealing were described in [3]. 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 according 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 for study.

3. Results and discussion
The X-ray diffractograms of the root and feather of the IN738 gas-turbine blade after regenerative annealing are presented in figure 1. In the sample cut from the blade root (Figure 1a), in addition to the $\gamma$-phase and $\gamma'$-phase lines, diffraction lines of MC and $M_{23}C_6$ carbides were found. The diffraction lines of the $M_{23}C_6$ carbide and the topologically close-packed (TCP) $\sigma$-phase were found in the sample cut from the blade feather (Figure 1b). This part of the blade usually operates at high temperature and is more stressed.

![Figure 1. X-ray results of the IN738 blade: a- the root; b- the feather.](image)

Structures of the root and feather of the blade are presented in figure 2. As can be seen from the figure 2a, carbides are observed inside the grains and on the grain boundaries of the sample cut from the blade root. Intergranular carbides have a well-defined globular shape of MC-type carbide. Carbides on the grain boundaries have a globular shape of MC-type carbide and an elongated shape which is typical for $M_{23}C_6$-type carbide. Figure 2b shows the results of optical microscopy of the sample cut...
from the blade feather. One can also see carbides inside the grains and on the grain boundaries. The precipitations of the irregular shape distributed along the grain boundaries and inside grains are associated with the σ-phase. The grain boundaries are completely covered with secondary M<sub>23</sub>C<sub>6</sub>-type carbides.

Figure 2. Microstructure of the root (a) and feather (b) of the blade, optical microscopy.

The SEM structures of the samples cut from the different parts of the studied blade are shown in figures 3-4.

Figure 3. Microstructure of the M<sub>23</sub>C<sub>6</sub> carbide (a) and MC carbide (b) in the blade root; the microstructure of the M<sub>23</sub>C<sub>6</sub> carbide (c) and MC carbide (d) in the blade feather.

The EDS analysis supports these observations (Table 1-2). Sigma phase is enriched with nickel, tantalum, titanium, niobium, and molybdenum. MC-type carbide in the sample cut from the blade feather contains less tantalum content than that of the carbide MC-type in the blade root. EDS analysis of the primary MC carbide shows a high content of titanium, niobium, and tantalum; chromium is practical absent. Secondary carbide M<sub>23</sub>C<sub>6</sub> has a high content of nickel, chromium, and cobalt. Grain boundary M<sub>23</sub>C<sub>6</sub> carbides in the blade feather have approximately the same chemical composition as that in M<sub>23</sub>C<sub>6</sub> carbides of the blade root.
Figure 4. EDS spectrum (a) and the structure (b) of the \(\sigma\)-phase in the blade feather, SEM.

Table 1. Chemical composition of the phases in the blade root, wt. %.

| Phases       | Ni  | Cr  | Co  | Mo  | Ta  | Al  | Ti  | C   | Zr  | Fe  | Nb  | W   |
|--------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Gamma prime  | balance | 13.66 | 7.42 | 1.67 | 4.93 | 2.49 | 3.67 | 2.02 | 0.89 | 0.47 | 1.70 | 2.06 |
| MC carbide   | balance | 1.42  | 0.95 | 1.60 | 46.07 | 0.23 | 24.14 | 1.92 | -   | 0.72 | 13.94 | 5.15 |
| \(M_23C_6\)  | balance | 14.96 | 7.84 | 2.34 | 2.83 | 2.00 | 3.65 | 4.06 | -   | 0.65 | 0.73 | 1.79 |

Table 2. Chemical composition of the phases in the blade feather, wt. %.

| Phases       | Ni  | Cr  | Co  | Mo  | Ta  | Al  | Ti  | C   | Zr  | Fe  | Nb  | W   |
|--------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Gamma prime  | balance | 14.40 | 7.93 | 3.16 | 5.14 | 2.01 | 3.40 | 1.47 | 0.81 | 0.62 | 1.27 |
| MC carbide   | balance | 2.32  | 0.92 | 14.79 | 21.06 | 0.52 | 22.59 | 1.53 | 2.69 | -   | 19.07 |
| \(M_23C_6\)  | balance | 13.62 | 7.72 | 2.97 | 4.32 | 2.27 | 4.36 | 1.36 | 0.82 | 0.22 | 2.50 |

It is known that \(M_23C_6\) carbides in nickel super alloys are mainly deposited from the matrix during heat treatment and service (at 760–980 °C) [2]. \(M_23C_6\) 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 [2]. According to the literature, hardening of the grain boundaries in heat-resistant nickel superalloy 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 μm 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 \(M_6C\) or \(M_23C_6\) carbides, leading to softening or embrittlement the alloy, should be minimized [3]. The type of carbide transformations and TCP phases depends on alloy doping and operating temperature. According to the literature, \(M_23C_6\) carbides and \(\sigma\)-phase may form in the IN738 super alloy after the exploitation for a long time at high temperature [6, 9]. The appearance of \(\sigma\)-phase as well as \(M_23C_6\) carbides lead to embrittlement the alloy. This process also points to the degradation of the alloy composition.

As can be seen from the Tables 1-2, the chemical composition of the gamma prime phase is different in the different parts of the blade. Comparison between Table 1 and 2 shows that the aluminum
concentration of the gamma prime phase which is responsible for hardening in the blade feather is decreased in compare with that in blade root. Such decrease in aluminum content testifies the degradation process in the blade. The process of redistribution the chemical elements in the alloy may be explained by the Gorsky effect which was observed previously in the gas turbine blade after long service time in [5]. 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. In our case this process may occur because 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 root part of the blade which is subjected the thermal load only. Service induced MC to M$_{23}$C$_{6}$ carbide transformation and formation of TCP σ-phase also indicate the redistribution of the chemical elements inside the material of the blade feather.

**Conclusion**

The following conclusions can be done from this study:

1. Phase transformations in the Inconel 738C alloy after standard regenerative heat treatment are studied. It is found the degradation of microstructure and chemical composition of the different parts of the IN738 gas turbine blade. It is suggested that the degradation process is caused by stress and high temperature.
2. Service induced MC to M$_{23}$C$_{6}$ carbide transformation and formation of TCP σ-phase indicate the redistribution of the chemical elements inside the material of the blade feather.
3. It is found that the standard regenerative heat treatment of the IN738 operative gas turbine blade does not effect on carbides transformation, TCP σ-phase dissolution, and thus do not guarantee the full recovery of the IN738 gas turbine blade.

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