REGENERATIVE HEAT TREATMENT OF BLADES OF HIGH-TEMPERATURE NICKEL ALLOYS

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An analysis of the operation of blades of gas turbine engines (GTE) manufactured from cast nickel alloys has made it possible to determine some of the causes of failure in operation, such as changes in the geometric dimensions or degradation of the microstructure. As a rule, the first cause is considered to be decisive. In order to increase the service life of blades partial regenerative repair of them is envisaged. In this case the dimensions of the blades are usually restored without allowing for possible changes in their structure, which can have a negative effect on operation after the restoration. For this reason, the second cause can become decisive in the reduction of their service life. This work is devoted to investigating the regularities of the degradation of the structure and properties of the material of cast GTE blades made of a high-temperature nickel alloy of the ZhS6U-VI type under operating conditions. A structural criterion determining the operating capacity of the olades and the ultimate level of structural degradation are established. A regenerative heat treatment to be conducted after partial exhaustion of the operating resource accompanied by structural changes that occur within the critical range of the structural criterion is suggested.

We investigated the microstructure of blades after service and the regularities of the formation of such structures in specimens subjected to a thermal load imitating the operating conditions of the blades.

We studied the microstructure of blades of cast alloy ZhS6U-VI (0.13 - 0.2% C, 5.1 - 6.0% Al, 0.8 - 1.2% Nb, 2.0 - 2.9% Ti, 1.2 - 2.4% Mo, 8.0 - 9.5% Cr, 9.5 - 11.0% W, 9.0 - 10.5% Co, the remainder Ni) before and after 1600-h operation.

The operating temperature of the blades varied within $950 - 1050^{\circ}$ C, and the stresses (in different cross sections) were 20 - 250 N/mm². The microstructure of the alloy before operation of the blade consists of coarse grains of a γ -solid solution, intermetallic particles of a γ' -phase (Ni₃(Al, Ti)), a carbide phase, and a $\gamma - \gamma' - MC$ eutectic (Fig. 1*a*). In operation of the blades the structure of the alloy changes continuously, including the morphology and size of the strengthening γ' -phase and the carbide phase and the formation and growth of borders along grain boundaries and around carbides in the grain bulk (Fig. 1*b* - *d*). A microscopic x-ray spectral analysis showed that the grain-boundary borders consist of an intermetallic γ' -phase with an elevated content of Ti and a reduced content of Cr. In operation, the carbide phase undergoes a transformation by the equation [1]

$$MC + \gamma = M_6C + \gamma'$$

Needle carbides M_6C found in the material of the blades after service are enriched with Ni and W. The stoichiometric composition of these carbides is Ni₃W₃C. Needles of double carbides are either directly bonded with primary carbides or grow from the borders around them (Fig. 1c).

The degradation of the structure of the alloy during service affects negatively their mechanical and high-temperature properties. After a thermal load lasting 500 h the strength properties of the alloy are degraded by 20% and after 1600-h operation the time before failure in tests for long-term strength decreases by a factor of 2.

An investigation of the regularities in the formation of degraded structures in specimens subjected to a heat load imitating the operating conditions of the blades showed that an increase in the test temperature intensifies initiation and development of the needle carbide phase, coagulation of the strengthening γ -phase, and growth of grain boundary borders. After a 500-h hold at 1050°C (under a load and without it) the number of nuclei of carbide needles increases tenfold, their mean length doubles, and the width of the grain-boundary borders increases by a factor of 2.5 – 3 compared to a hold at 950°C.

The growth of the secondary needle carbide phase with time occurs in two stages (Fig. 2*a*), namely, (*l*) intense generation and growth of carbides (a hold for at most 300 h at 1050°C) and (*ll*) deceleration of their growth (a hold for 300 - 500 h at 1050°C). The size of the grain-boundary borders depends on the degree of carbide changes in the alloy and, consequently, on the quantitative parameters of the needle carbide phase. The growth of the needle carbides in stage *l* is accompanied by an increase in the width of the grainboundary borders (Fig. 2*b*), whereas the deceleration of the carbide transformation in stage *ll* decelerates the develop-



Fig. 1. Microstructure of the forward edge of a blade of ZhS6U-VI alloy: a) before service; b - d) after 1600-h service; a - c) × 6000; d) × 10,000.

ment of grain-boundary borders and even leads to partial dissolution of them. This relationship is caused by redistribution of the γ' -forming elements between the bulk and boundaries of the grains in thermal loading. The growth of the borders in stage *I* is accompanied by their enrichment with Ti, whereas the dissolution of the borders in stage *II* decreases the concentration of Ti and Al in them. The concentration of Al decreases more rapidly than that of Ti, which increases the ratio Ti/Al (Fig. 2c) and strengthens somewhat the grain-boundary borders.

The growth of grain-boundary borders and the increase in the concentration of γ' -forming elements in them in stage *I* are accompanied by a decrease in the volume fraction of the γ' -phase in the grains (Fig. 2*d*). The tendency toward a decrease in the volume fraction of the strengthening γ' -phase in the grain bulk is maintained as long as the carbide reactions occur intensely, i.e., in stage *I*. The deceleration of carbide transformations in stage *II* is accompanied by growth of the volume fraction of the γ' -phase in the grains, which is associated with the process of partial dissolution of grain-boundary borders and the flow of γ' -forming elements from the boundaries into the grain bulk. This process occurs with strengthening of the internal volumes of the grains.

Thus, under operating conditions all structural components are transformed in the material of GTE blades, and the transformations are interconnected because they are caused by diffusion processes. The established regularities of the structural changes occurring under the action of a thermal load that imitates the operating conditions allow us to develop regimes for thermal regeneration of the structure, but what structural component is the weakest and therefore re-



Fig. 2. Length of needle carbides $l_c(a)$, width of grain-boundary borders d(b), titanium/aluminum ratio in the borders (c), and volume fraction of the γ' -phase V_{γ} in grains (d) as a function of the time of action of the thermal load at different temperatures and stresses: 1) 950°C, $\sigma = 0.9\sigma_{\tau}^{950°C}$; 2) 1050°C, $\sigma = 0.9\sigma_{\tau}^{950°C}$; 3) 1000°C; 4) 1000°C; 5) 1050°C.



Fig. 3. Microstructure of alloy ZhS6U-VI after the action of a thermal load at different temperatures: a) 950°C (a pore on a grain boundary); b) 1050°C (pores in the bulk of a grain); a) \times 1000; b) \times 500.

sponsible for disruption of the alloy in operation still remains unclear, whereas it should be the object of the regeneration. The material of the GTE blades works under conditions of elevated temperatures and moderate stresses and passes all disruption stages typical for creep, namely, initiation and development of pores and cavitation cracks, development of a main crack, and failure. It has been established that the pores appearing in the alloy can be classified into two groups according to their morphology, size, and place of initiation, namely, (1) pores initiating in grain-boundary borders (Fig. 3a) and (2) pores initiating in borders of globular carbides and the matrix (Fig. 3b). In the latter case they are finer and have a regular spherical shape.

Under the action of a thermal load at 950°C pores appear predominantly along grain boundaries in grain-boundary borders; at 1050°C they appear in the grain bulk. This regularity is natural and is caused by the special features of the structural state formed in operation of the blades and by the differences in the deformability of the grain boundaries and the grain bulk. The deformability of the grain bulk and the grain boundaries in such structures at the operating temperatures is



Fig. 4. Relation between local deformations of microvolumes ε_i ($d_{i0} \cong 50 \,\mu\text{m}$) and the relative elongation of regions δ_j ($l_{j0} \cong 500 \,\mu\text{m}$) for alloy ZhS6U-VI: *a*) after the action of a thermal load at 950°C for 500 h, $\sigma = 120 \,\text{N/mm}^2$, and extension at 950°C at a rate of $3.3 \times 10^{-4} \,\text{sec}^{-1}$; *b*) after the action of a thermal load at 1050°C for 100 h, $\sigma = 80 \,\text{N/mm}^2$, and extension at 1050°C for 100 h, $\sigma = 80 \,\text{N/mm}^2$, and extension at 1050°C at a rate of $3.3 \times 10^{-4} \,\text{sec}^{-1}$; *l*) grain-boundary microvolumes; 2) bulk of the grains.

evaluated by the method of temperature microscopy [2] with extension of the specimens at a rate of $3.3 \times 10^{-4} \text{ sec}^{-1}$, which corresponds to accelerated creep.

We established that after lengthy action of a thermal load at 950°C the bulk of a grain is deformed but little (by about 1.5%) whereas local regions with grain boundaries are deformed by more than 100% (Fig. 4*a*). The main role in the extension of a specimen is played by microcracks that appear on grain boundaries in the extension process. In early deformation stages fine spherical pores appear in grain-boundary borders. Having attained a certain size in grain-boundary borders positioned normal to the action of the tensile stresses, the pores merge by breaking the interpore bridges and form grain-boundary cracks. This mechanism of disruption is typical for blades under operating conditions. The grain boundaries are weakened by the grain-boundary borders, whereas the properties of the bulk of the grains remain virtually unchanged.

After lengthy action of a thermal load at 1050°C the deformation in repeated disruption develops in another way. The local deformation is uniform both in the grain bulk and along the grain boundaries and amounts to about 60% (Fig. 4b). The role of microcracks in the overall elongation is small, and grain-boundary borders do not become sources of disruption. This is so because the structure appearing in the grain bulk at 1050°C and under stresses applied for a long time increases the deformability of the grains manifold and causes formation of pores of type 2 predominantly in the bulk of grains and not along their boundaries, despite the presence of grain-boundary borders. This is a result of ordering of the particles of the γ' -phase in the process of their high-temperature coagulation under the stresses.

The particles forming a so-called quasi-periodic modulated structure [2] are ordered as a result of their growth in the predominant crystallographic direction with formation of flakes of a certain length. The ductility of the alloy increases in the ordering process by a mechanism that consists in that the density of the particles of the γ' -phase in the solid solution decreases considerably, whereas the mean free path of the dislocations increases much more than in the case of a random arrangement of the particles.

The coefficient describing the size ordering of the particles of the γ' -phase [3] is determined by the formula

$$k=A\,\frac{l\cdot b}{\delta}\,,$$

where *l*, *b* are the length and width of the flakes of the γ' -phase, μm ; δ is the distance between them, μm ; *A* is a coefficient describing the difference in the dispersity of the flakes of the γ' -phase, $1/\mu m$. We established that this coefficient correlates with the ductility level of the alloy. The higher the coefficient the higher the ductility of the alloy and the deformability of the bulk of the grains, and the less significant the role of grain-boundary borders as sources of disruption.

However, under actual operating conditions quasi-periodic modulated structures form only in high-temperature zones of the blades, which are not critical from the standpoint of disruption. The determining factor in the dangcrous zones of the blades after operation is a structure with particles of the γ' -phase randomly positioned in the bulk of the grains and grain-boundary borders, which weaken the grain boundaries and decrease the operating capacity of the blade as a whole. Grain-boundary borders embrittle the alloy only after attaining a critical size. An analysis of the effect of the size of the grain-boundary borders on the strength and ductility of alloy ZhS6U-VI showed that borders with a width below $2 - 2.5 \,\mu m$ hardly affect the mechanical properties of the alloy (Table 1). As the width increases to $3-4 \,\mu\text{m}$, the ductility characteristics decrease below the critical level, and the disruption becomes more intense. When the width of the borders exceeds the critical value, the part can fail and cause an emergency situation.

Thus, the main structural factor decreasing the service life of the blades is the presence of grain-boundary borders with a width exceeding 2.5 μ m. In order to restore the operating capacity of the material the borders should be eliminated in a timely manner, before the appearance of the porosity. A regenerative heat treatment should be conducted after evaluating the degree of degradation of the structure in certain zones of the blades using the width of the grain-boundary borders as the determining criterion.

According to the method of [4], regeneration of blades of an aircraft GTE made of alloy ZhS6U-VI is expe-

dient after the material has served for 40 - 50% of its resource time. At this point the structural changes in the alloy are considerable although the porosity does not affect its properties yet.

An investigation of alloy ZhS6U-VI after the action of a thermal load at 1050°C for 300 h showed that its endurance is reduced by 50%. The grain-boundary borders attain a maximum size and the pores in them have not yet formed. Specimens of the alloy subjected in this regime then underwent a regenerative heat treatment with the aim of eliminating the grain-boundary borders first of all. The specimens were held at 1210, 1215, 1225, or 1235°C for 4 h with cooling in air.

After a 4-h hold at 1210°C (a standard heat treatment) the streaks in the alloy remained completely intact and the strengthening γ' -phase in the bulk of the grains was partially dissolved. Heating to 1215°C restored the morphology of the γ' -particles inside the grains to a larger degree but the grain-boundary borders dissolved only partially; coarse particles of the γ' -phase remained in the structure, predominantly along grain boundaries. After a hold at 1225°C the grain-boundary borders dissolved completely and the morphology of the γ' -phase inside the grains was restored. Similar results were obtained after a hold at 1235°C.

Thus, regenerative heat treatment should be conducted at a temperature exceeding 1225°C for a time sufficient for dissolution of the grain-boundary borders. The hold time depends on the width of the borders. Timely elimination of continuous grain-boundary borders decelerates the formation of grain-boundary pores and cracks, preventing untimely disruption of the blades.

The efficiency of regenerative heat treatment is confirmed by results of an investigation of specimens cut from blade tongues after 600-h operation under severe conditions. The regenerative heat treatment was conducted by a regime that included heating to 1225°C for 4 h and cooling in air. This treatment restored the mechanism of deformation and disruption of the alloy under conditions of repeated extension at a rate of 3.3×10^{-4} sec⁻¹. At 950°C the intercrystallite disruption typical for the alloy after service was changed to the transcrystallite mechanism typical for the initial state. After the regenerative heat treatment the magnitude of the deformation due to development of grain-boundary cracks (δ_{cr}) decreased and the strength properties ($\sigma_{\rm b}$) were restored. The nonuniformity of microdeformations also fell to the initial level, which was indicated by the change in the standard deviation S of the local deformations ε_{n} from the mean value ε_{m} (Table 2).

| Treatment of the alloy | Width of borders, µm | σ_b^{ref} , N/mm ² | δ, % | Type of disruption | |
|---|-------------------------|--------------------------------------|-----------|--------------------|--|
| Initial state | 0 | 510±5 | 5.0 ± 0.5 | transcrystallite | |
| 950°C, 500 h | 2.5 | 525 ± 5 | 8.2 ± 0.5 | transcrystallite | |
| 1050°C, 500 h | 4 0 | 500 ± 5 | 1.5±0.5 | intercrystallite | |
| 950°C, 100 h, $\sigma = 180 \text{ N/mm}^2$ | 2.0 | 490 ± 5 | 5.0 ± 0.5 | transcrystallite | |
| 950°C, 300 h, $\sigma = 150 \text{ N/mm}^2$ | 3.0 | 460 ± 5 | 3.0 ± 0.5 | intercrystallite | |
| 950°C, 500 h, $\sigma = 120 \text{ N/mm}^2$ | 4.0 | 400 ± 5 | 3.0 ± 0.5 | intercrystallite | |

TABLE 2

| State of the alloy | σ_b , N/mm ² | δ _{cr} , % | S |
|--|--------------------------------|---------------------|------|
| Initial | 475 | 2.99 | 3.10 |
| After 600-h operation | 460 | 7.35 | 7.32 |
| A hold at 1225°C for 4 h, cooling in air | 495 | 1.47 | 2.77 |

Regenerative heat treatment.

Note. The presented properties of alloy ZhS6U-VI were obtained at $t_{\text{test}} = 950^{\circ}\text{C}$

The crack development $\boldsymbol{\delta}_{cr}$ was determined by the formula

$$\delta_{\rm cr} = \frac{\sum_{i=1}^{n} b_i}{l_0},$$

where $\sum_{i} b_{i}$ is the total width of cracks along the long axis of

the specimen, l_0 is the calculated initial length of the working zone of the specimen.

Evaluation of this characteristic shows that the number and size of grain-boundary cracks formed in the extended material decreases and strengthening of the grain boundaries occurs.

Long-duration tests of specimens cut from blades after operation for 1400 h that were conducted at a temperature of 975°C under a stress of 230 and 255 N/nim² showed that the regenerative heat treatment increases the service life of the specimens by 30 - 40%. After service the specimens failed in 40 and 22 h, and after the heat treatment the time before failure increased to 60 and 28 h, respectively.

Thus, heat treatment conducted in the stage of structural changes during operation when grain-boundary borders have already appeared but no pores have developed in them yet promotes effective regeneration of the structure and the mechanical and high-temperature properties of the alloy.

CONCLUSIONS

1. In cast high-temperature nickel alloys operation causes structural degradation, leading to the appearance of coarse structural components, namely, grain-boundary borders of a γ' -phase, whose number and size depend on the intensity of the carbide transformations.

2. A substantial contribution to the reduction of the service life of blades of gas turbine engines is made by the grainboundary borders, in which pores and cavitation cracks form during operation.

3. The width of the grain-boundary borders can be a quantitative structural criterion for evaluating the operating capacity of blades of cast high-temperature nickel alloys.

4. We have developed a regenerative heat-treatment regime that makes it possible to eliminate the degraded structure provided that the changes that have occurred in it during operation do not cause pores or microcracks. The treatment restores the microstructure of the alloy and increases the service life of the blade material by 30 - 40%.

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