# Improvement of Operating Properties of Heat-Resistant Alloys by the Structure Optimization

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**Abstract.** Fasteners of aircraft gas turbine engines are made of heat-resistant steels and alloys and are operated in intensive conditions under the action of high temperatures. For ensuring reliability, the connecting elements of aircraft engines are made of heat-resistant alloy steels. But under the influence of an aggressive environment, these elements are destroyed, resulting in problems during repairs. The structure of the material of fasteners during the threaded connection by cutting and plastic deformation is considered. The concentration of stresses in the thread plane depends on the density of dislocations, and the depth of corrosion penetration on the outer surfaces of the thread reaches 100 µm. It is proposed to introduce additional heat treatment of parts to improve the physical and mechanical characteristics of the alloy. After additional heat treatment, the σ-phase plates dissolve and prevent the development of micro-cracks. It is proved that the density of material dislocations in the state of delivery reaches critical values, but the introduction of additional heat treatment allows to increase the performance of parts made of alloy HX – Alloy Type 66Ni-17Cr regardless of the method of threading. Prerequisites for the rational selection of heat treatment modes have been developed, which provide the best mechanical and technological characteristics and the necessary structure of the materials used.

**Keywords:** Alloy · Deformation · Engine · Fractogram · Load · Resource · Wear resistance

## 1 Introduction

Threaded and riveted connections of parts, assemblies, and units of various types of equipment, including aircrafts [1] and engines [2] are the most common and reliable. Fasteners of gas turbines of aircraft engines work in particularly stressed conditions under static, dynamic, and vibration loads. As a result, these parts are subject to increased reliability requirements [3].

To increase aircraft engines' economic and operational performance, use various technological techniques that reduce the size of the relevant components, such as combustion chambers, but increase the load on the parts and, consequently, reduce their


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durability. Gas flows at the entrance to the turbine have a temperature of about 1500°K. Therefore, during operation, the damage of the surface layers from gas corrosion and the action of thermal and mechanical factors increases significantly [4]. Traditionally, the fasteners of gas turbine engines of aircraft are operated for one maintenance period, and to ensure the required resource, they are made of complex alloy heat-resistant steels and alloys such as follows: HD – Alloy Type 28Cr-6Ni; HN – Alloy Type 25Ni-20Cr; HX – Alloy Type 66Ni-17Cr; HW – Alloy Type 60Ni-12Cr; HX – Alloy Type 66Ni-17Cr [1]. The studied steels and alloys belong to the group: Heat Resistant Steel and Alloys ASTM A297 (Specification for Steel Castings; Iron-Chromium and Iron-Chromium-Nickel. For general application/ ANSI/ASTM A297/A297M-97) [5]. However, during the repair, there are some difficulties in disassembling such fasteners because the surface layers of the working surfaces of such parts have various types of damage [6]. Therefore, it is essential to solving the problem of increasing heat resistance and durability, reducing the level of various types of damage to parts made of heat-resistant steels and alloys [7].

## 2 Literature Review

The yield strength for the test material is 750 MPa. Thus, in the state of supply, the internal stresses exceed the yield strength, which can lead to relaxation processes, during which the deformation of the grains can cause microcracks [8]. After high-temperature heating, the internal stresses are less and do not exceed the yield strength, and the possibility of microcracks disappears or is significantly reduced [9]. In addition, it can be assumed that as a result of additional heat treatment [10], it is possible to eliminate existing microcracks [11].

The microstresses of the second kind, which occur during plastic deformation, are balanced in the volumes of individual crystallites (or blocks), associated with the inhomogeneous elastic deformation of crystallites and an inhomogeneous change interplanar distances [12]. In [13, 14], it was shown that dislocations lead to changes in interplanar distances, while impurity atoms and point defects, due to the relaxation of the lattice around them, do not make a significant contribution to the effect of the expansion of lines on radiographs.

The particles of the secondary phases released from the supersaturated solid solution significantly strengthen the alloy [15]. The increase in the yield strength $\sigma_T$ depends on the strength, structure, size, shape, distribution method, distance between the released particles, and degree of coherence of the particle lattices and the matrix [16]. All these factors affect the interaction of dislocations with the particles of the secondary phases [17]. In this case, there may be close interaction and long-distance effects associated with stresses caused by particle lattices and matrices [18].

## 3 Research Methodology

To solve the increasing heat resistance and durability of fasteners of aircraft gas turbine engines, parts (Fig. 1) of the most widely used material HX – Alloy Type 66Ni-17Cr were studied, mechanical properties of which are given in Table 1. As is known, this alloy belongs to heat-resistant [19].

a) b) c)

**Fig. 1.** General view of the surfaces of the fasteners of the aircraft engine: a) the surface of the part that has not been used; b) the surface of detail, after the exploitation with traces of various damages; c) the fracture surface of the threaded connection.

X-ray diffraction analysis of the samples was performed to assess the substructural state of the alloys for different methods of threading and, after the operation, additional heat treatment of parts. The DRON-3M unit was used for such studies. For determining the macro stresses in the test material, the X-ray diffractometric method was chosen, based on the exact determination of the deformation of the crystal lattice, because this express method has the following advantages: it is non-destructive and non-contact [20].

**Table 1.** Mechanical characteristics of HX – Alloy Type 66Ni-17Cr alloy.

| Material | $\sigma_T$, MPa | $\sigma_B$, MPa | $\sigma_B^{750}$, MPa | δ, % | ψ, % |
|---|---|---|---|---|---|
| HX – Alloy Type 66Ni-17Cr | 72 | 750 | 1150 | 17 | 19 |

Fractographic studies were performed using a scanning electron microscope REMMA-102A at a magnification of 120 to 1.2k times, which provides a significant depth of field (about 0.5 mm) and sufficient resolution (about 100…200 Å), which allowed to detect irregularities break and see all the details of the phase composition of the upper layers and identify the inclusion or selection [21]. The samples were cut from the destroyed fasteners taken in the state of delivery, after their operation, and after additional heat treatment, the elements of the destroyed surface were also examined (Fig. 1c).

## 4 Results

Characterizing the mechanical characteristics of the alloy HX – Alloy Type 66Ni-17Cr, according to Table 1, it is clear that this material has high values of $\sigma_B{}^{750}$, δ, ψ, which is an assessment of its operational reliability. However, under the action of high temperatures, such mechanical properties [22] as elasticity, yield strength, and strength are significantly reduced. Therefore, even at stress values that are less than the yield strength, the phenomenon of creep can occur, which is manifested in a gradual increase in plastic deformation. Since heat-resistant steels and alloys operate in a complex stress state (tension, compression, bending, torsion), combined with alternating vibration loads, these materials must resist fatigue at high temperatures and have high creep characteristics at normal temperature conditions [23].

Fasteners (bolts, studs, spokes) are made by cutting, and therefore the materials must be well subjected to this type of processing. However, it is known that cutting, including threading, heat-resistant steel parts, and alloys, is associated with significant difficulties. This primarily concerns the provision of the necessary roughness and the elimination of various types of damage to work surfaces during cutting in the form of material tears and burrs. Therefore, in this case, the thread on the fasteners is usually applied by rolling [1], which contributes to the appearance of a perfectly smooth surface that has sufficient depth and a strictly periodic step. This design allows you to securely fasten the connected parts and provide the necessary tightening force. However, this technology of cutting causes significant damage to the surface structure of the metal. Plastic deformation in the material when applying the thread promotes periodic zones with tensile and compressive stresses with increased dislocation density ($\approx 1 \cdot 10^{12}$ cm$^{-2}$). The possibility of forming and propagating cracks in the cut made by the rolling method is more significant than in the manufacture of threads by cutting [20]. The results of studies of the material HX – Alloy Type 66Ni-17Cr, conducted using an X-ray machine DRON-3M in iron radiation, show that the density of dislocations during rolling is more than twice as high as the density of dislocations during cutting. The concentration of stresses also depends on the density of dislocations, which significantly increases along the entire plane of the thread compared to non-working surfaces. It is known that the stress concentration for plastics materials is harmful under variable loads, elevated temperatures, and vibrations. Peak stresses near the concentrator can exceed the allowable resistance of the material under variable load, which leads to premature failure. The complex operating conditions of the gas turbine engine contribute to the emergence of various diffusion processes that adversely affect the fasteners of the alloy HX – Alloy Type 66Ni-17Cr. In particular, in places of high-stress concentration, microcracks often occur, which penetrate the active components from the environment and can cause a wedging effect (Rebinder effect), the development of corrosion, and other negative processes that significantly affect the durability of such parts and can cause significant difficulties disassembly of the gas turbine engine. This type of brittle fracture occurs under the action of surface-active or corrosive media due to overheating and when exposed to the material of other molten metals.

Appropriate protective coatings are often used to protect against corrosion and various diffusion processes. However, this approach complicates the technological process of manufacturing fasteners and does not always lead to the desired result. In addition, when tightening the fasteners, the protective coatings can be damaged while facilitating the diffusion of active gases from the environment due to the accumulation of dislocations in the places of the cut. The fracture surface of an emergency part is usually contaminated with soot, grease, corrosion products, or oxide film, the formation of which is associated with the action of high temperatures on the part (Fig. 1b). Analysis of the surface layer structure shows that the depth of corrosion penetration on the outer surfaces of the threads operating in the zone of high temperatures reaches 100 μm. In some cases, it was found that the separation of oxide films can occur. Such damage at the mentioned loads is the main reason for the development of fatigue failure. The fractograms of HX – Alloy Type 66Ni-17Cr alloy in the state of delivery and after hardening and aging (Fig. 2) show signs of brittle fracture in the form of a chip near the thread with a gradual transition to intergranular fracture with increasing distance from the thread surface.

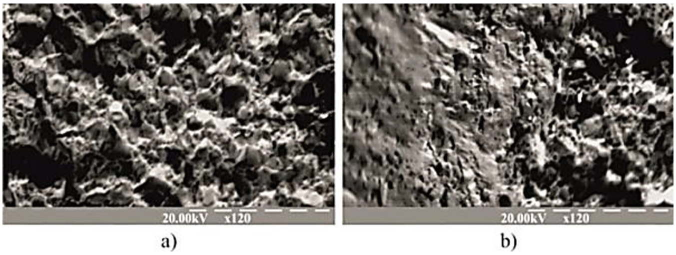



**Fig. 2.** Fractogram of HX – alloy type 66Ni-17Cr alloy in the state of delivery (×120): a) in the center of the sample; b) near the cut.

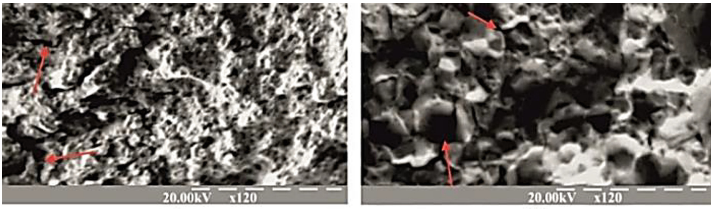
Figure 2 clearly shows the signs of hollow fracture and fracture along the grain boundaries of the alloy HX – Alloy Type 66Ni-17Cr, visible microcracks located at the boundaries of the inclusions.

In the study of these same samples after heat treatment at 900 °C for 20 min, a brittle intergranular fracture is observed (Fig. 3): at the grain boundaries weakened by carbide emissions (Fig. 3a) and at grain boundaries enlarged by coagulated emissions Fig. 3b).

In the structure (Fig. 3b), microcracks are visible, located along the boundaries of inclusions and microzones with a hollow structure.

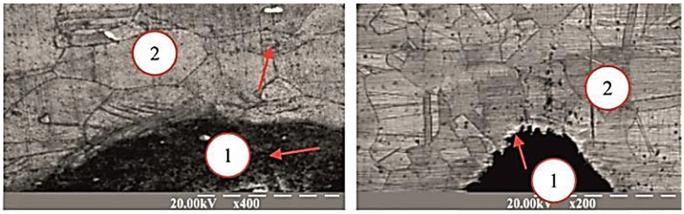
As deformation at the drawing of a carving (rolling or cutting) is conducted at low temperatures, in a near-surface layer, the slander is formed. Therefore, a fine-grained structure with thin-plate or fibrous grains is observed (Fig. 4a).

Changes also occur in the near-surface layer after conducting studies of the impact of operation ($\tau$ = 40…80 h). After the long-term operation at elevated temperatures, a fine-grained, partially recrystallized structure is observed (Fig. 4b) with signs of recrystallization texture.




a) b)

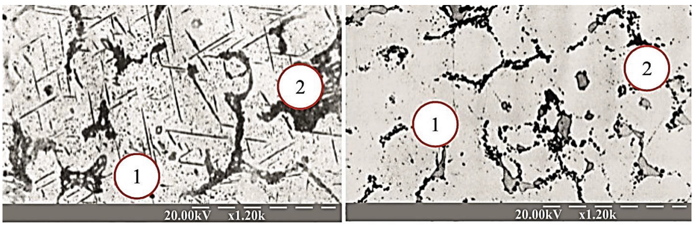
**Fig. 3.** Fractogram of alloy HX – Alloy Type 66Ni-17Cr after heat treatment at 900 ˚C (×120): a) near the cut; b) in the sample center.




a) b)

**Fig. 4.** The structure of the etched surface layer of the alloy HX – Alloy Type 66Ni-17Cr (1 – subsurface layer, 2 – base metal): a) before exploitation (×400); b) after exploitation (×200).

The surface formation of the alloys for an oxide layer is accompanied by a redistribution in the surface layer of the matrix of alloying elements and the formation of an intermediate zone in the form of an oxidized white zone. Alloying elements diffuse into the scale material. The formation of a layer depleted by the alloying element leads to a decrease in hardness, strength, reduction of the crystal lattice period, and the occurrence of stresses. The stresses at any temperature and cause the accumulation of defects and accelerate nucleation and development of cracks. In fasteners made of HX – Alloy Type 66Ni-17Cr alloy, which has worked less than the estimated technical resource, the metallographic analysis revealed σ-phase plates, which may be one of the causes of premature failure of parts (Fig. 5). For addressing this issue, additional heat treatment was performed. After additional heat treatment (1200 ºC, 20 min), the σ-phase plates dissolved and, accordingly, the plastic characteristics of the alloy significantly improved.




a) b)

**Fig. 5.** σ-phase in the alloy HX – Alloy Type 66Ni-17Cr (×1200); (1 – plates of the σ-phase in the main matrix; 2 – the base metal): a) after exploitation; b) after dissolution as a result of additional heat treatment.

Prolonged exposure to high temperatures (500…800 °C) during operation leads to the release of secondary phases – intermetallic compounds at the grain boundaries, making them part of the alloy brittle, significantly reducing their plastic properties and toughness. According to the analysis of the conducted research, σ-phase $(NiCo)_7(WMo)_6$ and μ-phase (NiCoCr)(WMo) are formed in chromium-nickel alloys and steels, due to which this embrittlement process is called "sigmatation". The intensity of the sigmatation process is determined by the intensity of diffusion processes, and therefore its duration at low temperatures of 500…600 °C is very significant (100…1000 h) and is significantly accelerated at temperatures of the order of 800 °C.

These phases are unstable. The elementary cell of the σ-phase crystal lattice consists of 30 atoms (Fig. 6).

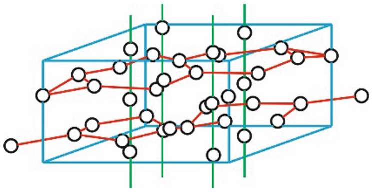


**Fig. 6.** General view of the σ-phase lattice.

These phases are formed on carbides. As a result of growth, they penetrate all the grain of the metal matrix. The lamellar shape of the phases contributes to the brittle destruction of the alloy. In addition, by extracting refractory metals from the solid solution, these phases weaken it. σ-phases are formed in the form of segregations with chromium carbides in nickel-chromium alloys with a high total content of Al and Ti (more than 10% by weight). The presence of these phases increases the fragility and reduces the ductility, especially under dynamic loads, and reduces the long-term strength at high temperatures, i.e., heat resistance.

Using X-ray diffraction analysis on the DRON-3M device, the details of the HX – Alloy Type 66Ni-17Cr alloy were investigated, and the crystal lattice parameter $\alpha$, the size of the X-ray scattering blocks D, the relative change of the lattice parameter $\Delta\alpha/\alpha$, the mean square displacement of atoms from the equilibrium position $\sqrt{\bar{u}^2}$ and density ρ were evaluated. In addition, the main component of the studied steels and alloys ($Fe_\alpha$ and $Fe_\gamma$) was evaluated. From the obtained results $Fe_\alpha \approx 5\%$, $Fe_\gamma \approx 95\%$. The measurement error in determining D and $\Delta\alpha/\alpha$ was 16…18%, the error in determining the density of dislocations did not exceed 30…40%. The crystal lattice period was calculated with an accuracy of 0.02…0.03% of the specified value. The research results are presented in Table 2.

From the results shown in Table 2, it is seen that the density of dislocations ρ after standard heat treatment, i.e., in the state of delivery, reaches almost a critical value of $10^{12}$ 1/cm$^2$ and depends little on the method of threading (cutting or thread rolling). This density of dislocations causes increased fragility of aircraft engine mounts in the state of delivery and is one of the reasons for their premature destruction. The dislocation density decreases slightly during operation because the operating temperature (850 °C) is lower than the recrystallization temperature (900 ºC). Further evolution of the dislocation structure is associated with the consolidation of dislocations due to the formation of Cottrell atmospheres during operation and the formation, development, and propagation of microcracks under static and alternating loads, which cause fatigue failure of parts.

**Table 2.** The results of X-ray-structural studies.

| Object of research | α, Å | D, sm | Δα/α | $\sqrt{\bar{u}^2}$, Å | ρ, $sm^{-2}$ | Remark |
|---|---|---|---|---|---|---|
| HX – Alloy Type 66Ni-17Cr | 3.5953 | $8.4 \cdot 10^{-6}$ | – | 0.11 | $0.14 \cdot 10^{12}$ | In the initial state |
| HX – Alloy Type 66Ni-17Cr | 3.5940 | $2.1 \cdot 10^{-5}$ | $0.18 \cdot 10^{-2}$ | 0.08 | $6.8 \cdot 10^{9}$ | After additional heat treatment |

This high density of dislocations can be explained by phase hardening due to the release of secondary strengthening phases during aging studied the metastable γ′-phase.

Therefore, it is evident that the additional heat treatment can significantly increase the performance of parts made of alloy HX – Alloy Type 66Ni-17Cr, regardless of the threading method.

## 5 Conclusions

To slow down the fatigue failure of parts and increase their durability, it is necessary to improve the structure of the surface layer of fasteners significantly. The creep of metals causes stress relaxation in pre-loaded parts (tightened fasteners gradually weaken). Therefore, the coarse-grained structure is unacceptable, as it provides increased sensitivity to stress concentrators, which will be the cutting of parts. The manufacture of parts should consider the appearance of zones with tensile and compressive stresses with high dislocation density, which depends on the stress concentration. It is necessary to carefully select the mode of heat treatment, which could provide better mechanical and technological characteristics and the required structure of the materials used. The performed research will be continued and serve to improve the technological process of manufacturing threaded parts from heat-resistant steels and alloys and improve the heat resistance of these materials by optimizing the heat treatment modes.

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