

## The Effect of Thermomechanical Treatment Conditions on the Structure and Properties of the Granulated EP741NP Nickel Alloy

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### Abstract

The regularities of the influence of temperature, velocity and degree of deformation, as well as heat treatment modes on the microstructure and properties of the EP741NP nickel alloy made by the hot isostatic pressing method are investigated. It has been shown that the heterogenizing annealing leads to the isolation of the  $\gamma$ -phase coagulated up to 1.1  $\mu\text{m}$  size of the  $\gamma'$ -phase precipitates with partially coherent interphase boundaries and creates the most favorable conditions for the development of recrystallization processes during subsequent hot strain. Optimal parameters of the thermomechanical treatment providing the formation of a homogeneous ultrafine-grained (UFG) microduplex structure with the given grain sizes of the  $\gamma$ -phase and incoherent particle grains of the  $\gamma'$ -phase in the deformed preform from the granulated EP741NP alloy are determined. It is shown that the EP741NP alloy in the UFG condition demonstrates high superplasticity (SP) properties. At a temperature of 1100°C and a strain rate of  $10^{-3}\text{s}^{-1}$ , the elongation was 630 %, and the velocity sensitivity coefficient  $m = 0.57$ . It is shown that by varying the heat treatment modes for the material with the UFG structure the microstructures with specified grain sizes of the  $\gamma$ -phase (from 10 to 67  $\mu\text{m}$ ) and coherent precipitates of the hardening  $\gamma'$ -phase can be formed; this will make it possible to achieve the required set of heat-resistant properties in the finished part. Thermomechanical treatment of the EP741NP alloy with the UFG structure involves its annealing under the conditions of a temperature gradient, subsequent hot strain and heat treatment in a two-phase region. It will make it possible to create a regulated gradient change of the microstructure along the radius of gas turbine engine disk (a fine grain microduplex bore, a “necklace” web and a coarse grain rim with tortuous grain boundaries) and, accordingly, ensure that the required functional gradient properties.

### Keywords

Superalloys; heat treatment; hot isostatic pressing method; heterogenizing annealing; ultrafine-grained structure; heat-resistant properties.

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### Introduction

Superalloys are widely used in production of aviation gas turbine engines (GTE) and similar power plants [1–3]. Traditionally, the problem of improving heat-resistant properties has been solved by creating more complicated superalloys compositions, but this has led to a sharp increase in the complexity of treatment of these alloys due to reduced technological plasticity and increased resistance to deformation. In addition, superalloys are characterized by a significant dendritic liquation and heterogeneity of chemical composition, because of which it is necessary to limit

the dimensions of ingots and deformed semi-finished products. The use of methods of powder metallurgy to produce superalloys eliminates many of these shortcomings [4]. In addition, the working properties of powder alloy parts are largely determined by the quality of the consolidated powder, which is difficult to control in bulk materials for a number of parameters, such as the degree of contamination and porosity. To reduce the negative effect of defects, reduce the size of granules, methods of control and cleaning of impurities are being improved. Another more effective way to increase the mechanical properties of powder alloys is to include hot strain in

the manufacturing process of the part. For example, in [5] the problems of using granulated superalloy in promising GTEs, using the experience of General Electric Company (the USA), were described. It was noted that in order to prevent hazardous defects formed as a result of contact with organic impurities, it is necessary to manufacture the preform disks by extruding powdered material and by subsequent isothermal forging. The results showing the favorable effect of hot strain on the structure and properties of the granulated EP741HP alloy are given in [6, 7]. Meanwhile, the problem of achieving the maximum technological plasticity of superalloys, as well as improving the quality of parts made from them and reducing labor intensity can be solved by developing new resource-saving technologies based on the superplasticity effect, a necessary condition for the realization of which is the formation of the UFG structure [8–10] in semifinished products.

Superalloys during operation are subject to a complex impact of temperature and loads. Therefore, in critical parts, for example, GTE disks made from both deformable [11] and powdered superalloys [12, 13], it is important to create a gradient microstructure with an optimal set of properties under the conditions of the temperature gradient of their operation. The research into possibility of purposeful changes in the structure of parts made from powdered superalloys along the cross-section (for example, in radial direction, from the hub to the rim, the most heated part of the GTE disk) is extremely relevant. Such changes can give the parts the optimum set of properties for operating conditions as such parts will be in demand for engines of the 5th generation.

This paper aims to study the regularities of the impact of deformation conditions and heat treatment on the processes of structure formation in the solid phase state in the powdered EP741NP alloy. These regularities will allow selecting optimal processing modes to achieve the required structural states and high level of technological (superplastic) and operational properties in semifinished products and components from this material.

### Materials and Methods

The industrial powdered EP741NP alloy obtained by hot isostatic pressing (HIP) was used as the research material. The investigated alloy had the following chemical composition (in wt%): Ni-base, Cr – 9.5; Ti – 1.8; Al – 5.3; Mo – 4.1; Nb – 2.7; Co – 16; W – 5.9.

To study the regularities of the effect of temperature, degree and strain rate on the microstructure and properties of the EP741NP alloy, the cylindrical workpieces sized  $\varnothing 10 \times 15$  mm were deformed by a uniaxial compression on a *Shenck Trebel RMS100* tester under isothermal conditions in a wide range of temperatures, which were at 10–285 °C below the temperature of complete dissolution of the  $\gamma$ -phase ( $t_s$ ). Since the EP741NP alloy in a coarse-grained condition after the HIP is characterized by low technological plasticity, to minimize the formation of cracks on the side surface of the samples, the sinking strain was performed fractionally in the temperature interval  $t_s = 10 - 45$  °C with intermediate annealing at the deformation temperature. Large workpieces sized  $\varnothing 90 \times 60$  mm were deformed on a press with a force of 630 tons using a heat-insulated container made of steel, the layout of which is shown in Fig. 1.

### Results and Discussion

*Formation of the UFG structure of the EP741NP alloy under thermomechanical treatment.* In the initial state, the alloy had a coarse-grained structure formed by sintering of the powder during the HIP (Fig. 2, a) and subsequent standard heat treatment of the EP741NP alloy. The average grain size of the  $\gamma$ -phase was  $d = 50$   $\mu\text{m}$ . The intragranular  $\gamma'$ -phase of predominantly cuboidal shape was uniformly distributed and coherent to the  $\gamma$ -grains of the matrix, as evidenced by the Moire-type banded contrast at the interphase boundary (Fig. 2, b).

To improve the deformability of the EP741NP alloy in a coarse-grained state, heterogenizing annealing (HA) was carried out prior to deformation with heating to a temperature of  $t_s - 10$  °C and subsequent cooling with a rate of  $\sim 25$  °C/hr to the aging temperature [14]. The analysis of the fine microstructure showed that after the HA, the distance between the  $\gamma'$ -phase precipitates ( $\lambda\gamma'$ ) increases due to the coagulation and coalescence of  $\gamma$ -phase particles (Fig. 2, c, Table 1). The dislocations that appear at the interphase  $\gamma/\gamma'$ -boundary indicate a partial loss of coherence at the interface between the matrix and the  $\gamma'$ -phase.

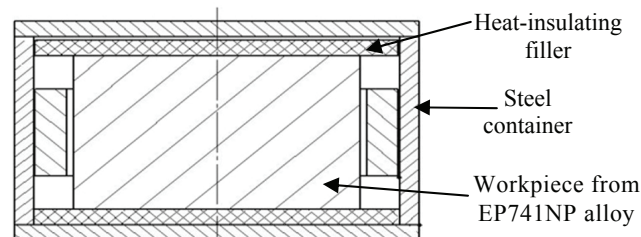


Fig. 1. Layout of container for thermomechanical treatment of EP741NP alloy

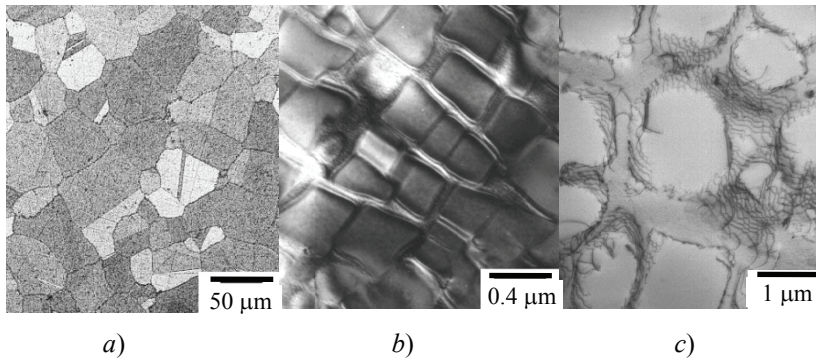


Fig. 2. Microstructure of the EP741NP alloy in the initial state: (a, b) and after the HA (c)

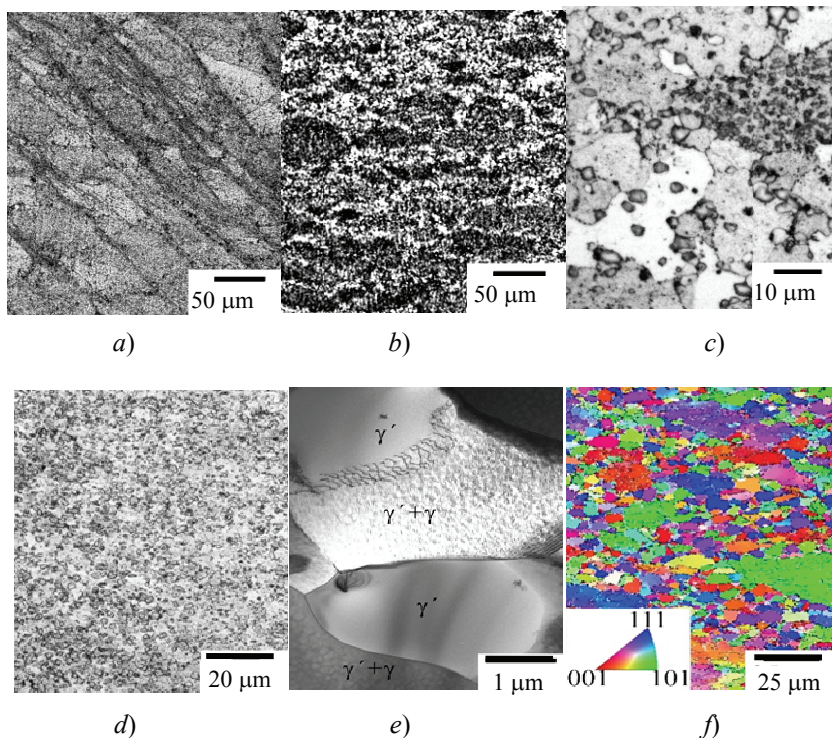


Fig. 3. Microstructure of the EP741NP alloy after a single strain of 70 % at temperatures:

$t_s = 285$  °C (a);  $t_s = 60$  °C (b);  $t_s = 25$  °C (c), and also after fractional strain of samples in the temperature range  $t_s = 25 - 40$  °C (d–f)

The analysis of the stress-strain curves obtained during the sinking strain showed that the preliminary HA led to a decrease in the level of the flow stress by 15–20 % compared to the level of stress at the alloy strain in the initial state.

The microstructure of the alloy after deformation in the investigated temperature range is shown in Fig. 3. In a one-stage strain by 70% at low temperatures ( $t_s = 185 - 285$  °C), deformation was localized in the lines oriented at an angle of  $45^\circ$  to the compression axis (Fig. 3, a), which led to the occurrence of deep cracks on the lateral surface of

the deformed samples. In the microstructure of samples deformed once at higher temperatures ( $t_s = 60 - 185$  °C), the deformation and recrystallization processes developed much more uniformly and a partially recrystallized microstructure was formed (Fig. 3, b). An increase in the deformation temperature ( $t_{def}$ ) led to an increase in the volume fraction of recrystallized grains ( $V_{recr}$ ). Thus, after a single deformation of 70 %  $V_{recr}$  increased from 30 % at  $t_{def} = t_s - 135$  °C to 50 % at  $t_{def} = t_s - 60$  °C.

Fractional strain of the samples with intermediate annealing at an elevated deformation temperature  $t_{def} = t_s - 10$  °C contributed to an increase in the volume fraction of recrystallized grains to 75 %. However, due to a sharp decrease in the volume fraction of the strengthening phase to 5–8 %, the  $\gamma$ -phase grains grew intensively to a size  $d_\gamma = 69$   $\mu\text{m}$  and a structure close to the matrix phase was formed. Based on the results of the experiments, it was established that the optimal UFG structure of the microduplex type is formed in the process of thermomechanical treatment, including fractional strain with a total

Table 1.

Effect of HA on the parameters of the EP741NP microstructure

State	$d_\gamma, \mu\text{m}$	$d_{\gamma'}, \mu\text{m}$	$\lambda_{\gamma'}, \mu\text{m}$	$V_{\gamma'}, \%$
Initial	50	0.4	0.10	$56 \pm 3$
After HA	50	1.1	0.8	$55 \pm 3$

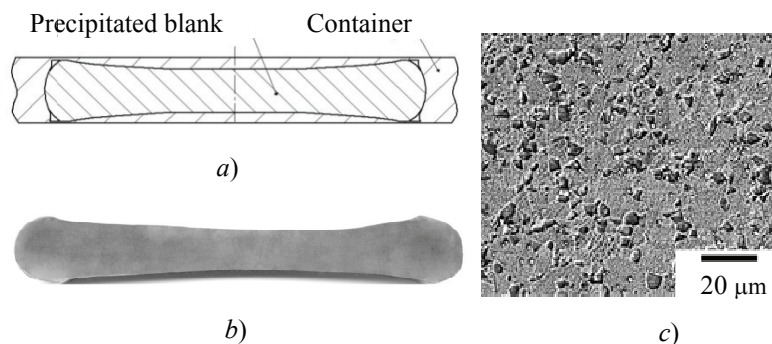


The obtained results indicate that in the process of thermomechanical treatment, the UFG structure is formed, consisting of grains of the  $\gamma$ -matrix and coagulated  $\gamma'$ -phase precipitates, which have predominantly incoherent high-angle  $\gamma/\gamma'$ -boundaries (Fig. 3, *d, e*). The EBSD method has shown that non-recrystallized regions are islands with a developed substructure (Fig. 3, *c*), in which the sub-grain size is commensurable with the interparticle distance between particles of incoherent  $\gamma'$ -phase precipitates.

Thus, the use of a preliminary high-temperature annealing in combination with fractional strain with post-deformation annealing in the temperature range  $t_s = 35 - 25^\circ\text{C}$  makes it possible to form a fine-grain microduplex structure in the powder EP741NP alloy. The size of the  $\gamma$ -grains in the microduplex structure depends on the deformation temperature, and the size of the  $\gamma'$ -phase depends on the heat treatment before deformation. The formation of such a microstructure is a necessary condition for the implementation of promising resource-saving technological processes based on the super plasticity effect, for example, in production of gas turbine engine disks [8, 15–17].

The proposed differential heat treatment modes were tested when large preforms sized  $\varnothing 90 \times 60 \text{ mm}$  were machined from EP741NP alloy under quasi-isothermal conditions using a heat-insulating container (see Fig. 1). The study of the macrostructure of deformed preforms has shown that stagnant zones are practically absent due to the use of a shell. After fractional strain combined with intermediate annealing, a homogeneous, recrystallized structure of the microduplex type was formed in the preforms (Fig. 4). The volume fraction of the recrystallized grains was  $\sim 80\%$ , the average size of the recrystallized grains of the  $\gamma$ -grains was  $5.5 \mu\text{m}$ , and the size of the incoherent  $\gamma'$ -phase precipitates was  $2\text{--}2.7 \mu\text{m}$ .

The larger size of the incoherent  $\gamma'$ -phase precipitates obtained by differential heat treatment of large preforms in comparison with similar treatment of small preforms is apparently due to a longer thermal decomposition effect on the structure of the EP741NP alloy. It should also be noted that the proposed conditions of thermomechanical treatment ensured a high yield of the usable metal due to minimization of the crack formation during deformation.



**Fig. 4. Diagram of a strained preform in the container (a) and macro- (b) and microstructure (c) preforms from the EP741NP alloy after differential heat treatment in quasi-isothermal conditions**

As was noted earlier in [8, 10], differential heat treatment followed by the SP deformation and final heat treatment favorably affects the mechanical properties and reliability of the GTE parts from nickel superalloys. Below we show the use of the powder EP741NP alloy as an example.

*Microstructure and properties of the EP741NP alloy after differential heat treatment and thermal treatment.* As is known [8, 10], after the SP deformation, the HRNAs with UFG structure must be subject to thermal treatment to recover their heat-resistant properties. In some cases, it is sufficient to perform standard heat treatment. Thus, for most of superalloys deformed by the SP method, a standard heat treatment with heating to a temperature equal to or exceeding the temperature of complete dissolution of the hardening  $\gamma'$ -phase, a coarse-grained structure with the required grain size is formed, and subsequent aging provides a homogeneous isolation within the coarse grains coherent particles of the strengthening phase. This ensures a complete recovery of the heat resistant properties that are required by the technical operating conditions, for example, gas turbine engine parts.

Heat treatment of the EP741NP alloy after the differential heat treatment (DHT) was carried out in 4 variants mainly by varying the quenching temperature, which made it possible to form structural states with different grain sizes of the  $\gamma$ -phase from  $10$  to  $67 \mu\text{m}$ . Such a difference in the size of the  $\gamma$ -phase grains, despite the fact that the aging modes were the same, had a significant effect on the mechanical properties of the EP741NP alloy. The results of mechanical tests and the microstructure of the EP741NP alloy after the differential heat treatment and heat treatment according to different modes are given in Tables 2 and 3, and in Fig. 5.

Table 2

**Short-term mechanical properties of the EP741NP alloy after differential heat treatment in the ( $\gamma+\gamma'$ )-region and heat treatment**

Heat treatment option	$T$ , °C	$d$ , μm	$\sigma_v$ , MPa	$\sigma_{0,2}$ , MPa	$\delta$ , %	$\psi$ , %	$a_n$ , MJ/m <sup>2</sup>	HB
1	20	10	1652	1122	23.1	15.3	0.29	426
			1644	1116	24.1	22.1		
	650		1404	1055	23.3	19.7		
			1062	24.4	21.0			
2	20	20	1588	1089	22.1	20.2	0.38	425
			1583		23.1	17.1		
	650		1434	1047	29.9	22.6		
			1397	1040	26.1	24.5		
3	20	48	1535	1120	21.6	19.2	0.57	417
			1534	1106	22.4	21.0		
	650		1020	23.7	20.3			
				1385	1025	23.6		
4	20	67	1545	1144	20.6	17.2	0.5	415
			1533	1112	21.0	17.8		
	650		1400	1019	20.4	19.2		
			1385	1024	23.7	18.9		
Material specifications	20	—	≥1421	≥1000	≥13	≥15	≥0.39	312 – 425

The analysis of the results showed that after the final heat treatment, the deformed material achieved a high level of mechanical properties as compared with the requirements for the EP741NP alloy, subjected only to hot isostatic pressing. It should be noted that samples in which a fine-grained structure was retained after the final maintenance (Fig. 5, *a*) showed the highest strength properties at 20 °C, while the alloy with a relatively coarse-grained structure (48  $\mu\text{m}$ , Fig. 5, *c*) showed the smallest elongation at rupture life tests. These results show the impact of deformation and heat treatment modes on the structure and properties of the EP741NP alloy, and are in good agreement with the results obtained in [7].

Thus, by varying the heat treatment modes, we can obtain higher heat-resistant properties and toughness or higher short-term strength in the deformed powdered EP741NP alloy.

Table 3

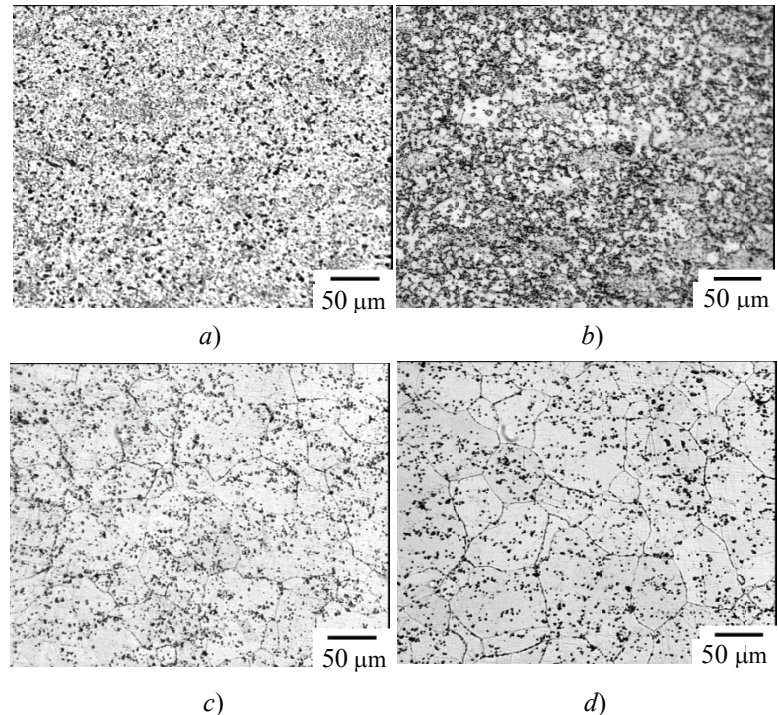
**Characteristics of long-term strength of the EP741NP alloy after thermal treatment under various modes**

Treatment mode		Applied stress and testing temperature $\sigma = 980 \text{ MPa}$ , $t = 650 \text{ °C}$		
Condition	Thermal treatment mode	Duration of the test, $h$	$\delta$ , %	$\psi$ , %
HIP	B	399	2.61	0.81
<i>Deformed state + thermal treatment</i>				
HIP+DHT	A	365*	2.50	1.60
HIP+DHT	B	367*	1.45	0.60
HIP+DHT	C		0.92	0.40
HIP+DHT	D	399*	1.83	1.40

\*No ruptures in samples, tests terminated

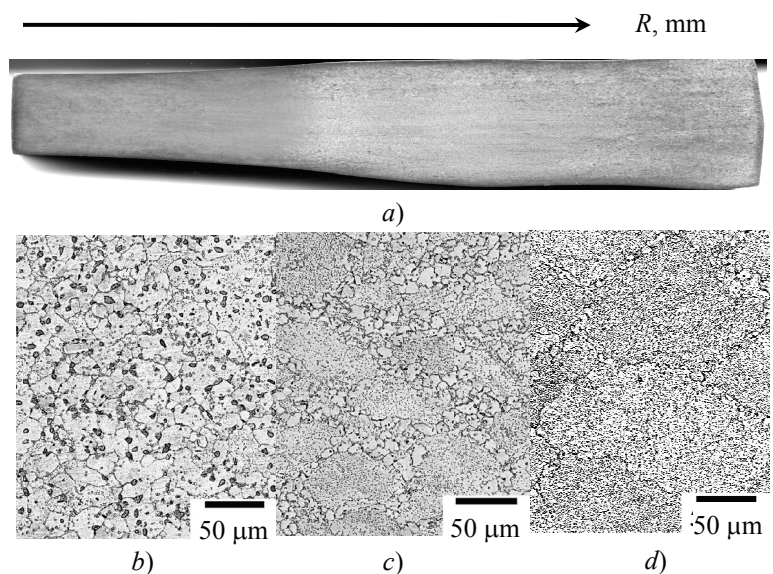


The effect of conditions of thermomechanical treatment on the formation gradient regulated structure in the GTE disk preform. The regularities of the effect of the differential heat treatment modes on the structure and properties of the EP741NP alloy were used to create a gradient structure in the deformed preform: a fine-grained structure in a hub, and a relatively coarse-grained “necklace” structure in the rim. To obtain a disk preform with a gradient structure, differential heat treatment was conducted in several stages. In the first stage of differential heat treatment, a heat-insulating container was used to produce a strained preform with a uniform ultra-fine structure throughout the entire section. Further, this preform was subjected to heat treatment under temperature gradient conditions by the method described in [8, 11, 18] and the analogous procedure given in [13]. As a result of this treatment, an ultrafine microduplex grain structure was preserved in the central part of the disk preform, and a coarse-grained matrix type structure with a grain size of  $\gamma$ -phase of 50–70  $\mu\text{m}$  was formed in the peripheral part. In the next step, the preform was subject to additional deformation in the two-phase region by a degree of 15–35 % and heat treatment at a temperature below the complete dissolution of the phase. As a result of such treatment, it was possible to form a radial gradient change in the microstructure over the section in the deformed preform of the disk (Fig. 6). As can be seen from Fig. 6, the central part of the preform has the UFG microduplex structure. Then, with an increase in the radius ( $R$ ) of the model disk preform to about  $0.3R$ , a transition zone from the UFG structure to the “necklace” type structure was observed; this is a coarse-grained structure with a chain of small recrystallized grains bordering large grains. It is important to note that with a further increase in the radius from  $0.4$  to  $1.0 R$ , there was a monotonous decrease in the volume of UFG structure in the “necklace” structure. As a result of this, a gradual transformation of the necklace structure into a coarse-grained structure with tortuous grain boundaries occurred, as was shown in [11]. In other words, instead of a chain of UFG grains, which practically disappeared at the periphery, the tortuosity of the  $\gamma$ -phase grain boundaries was formed due to the



**Fig 5. Microstructure of the EP741NP alloy after the differential heat treatment and heat treatment according to the different modes:**

*a* – mode 1; *b* – mode 2; *c* – mode 3; *d* – mode 4



**Fig. 6. Macro and microstructure of a model disc blank with a gradient regulated structure in the radial direction**

formation of subgrains in the border regions and local migration of the boundaries, as was shown in [11].

The analysis of the results of mechanical tests of samples cut from various zones of the model disk preform showed the following. The maximal values of strength ( $\sigma_{0.2} = 1136 \text{ MPa}$ ,  $\sigma_v = 1670 \text{ MPa}$ ) and plasticity,  $\delta = 23 \%$ ) were observed at room temperature in the central part of the disk hub, and

decrease somewhere in the rim part, in which ( $\sigma_{0.2} = 1040\text{--}1070$  MPa,  $\sigma_v = 1550\text{--}1570$  MPa,  $\delta = 18\%$ ). The values of the impact strength increased on the contrary with increasing radius of the attainable values in the disk rim,  $a_n = 0.5$  MJ/m<sup>2</sup>.

It should be noted that the research findings are in good agreement with the results of work [11], in which a deformable EP962 nickel alloy subject to differential heat treatment in the SP temperature-velocity mode had a similar change in the microstructure in the cross disk section, resulting in the improvement of high-temperature properties of the part. It was shown that due to the formation of the “necklace” and coarse-grained structure with tortuous grain boundaries in the web and rim of the disk, an additional sub-structural strengthening effect is ensured, which cannot be achieved only by gradient heat treatment, which was applied in the works [13, 19].

### Conclusions

The regularities of the impact of temperature, speed and degree of deformation, as well as heat treatment modes on the microstructure and properties of the EP741NP nickel alloy manufactured by the hot isostatic pressing method were investigated.

The optimal parameters of thermomechanical treatment ensuring the formation of a homogeneous UFG microduplex structure with given grain sizes of the  $\gamma$ -phase and non-coherent grains of the  $\gamma'$ -phase in a deformed preform made from the granulated EP741NP alloy are found.

It is shown that the EP741NP alloy in the UFG state demonstrates high superplastic properties. At a temperature of 1100°C and a deformation rate of  $10^{-3}$  s<sup>-1</sup>, the relative elongation was 630 %, and the velocity sensitivity factor  $m = 0.57$ .

It is shown that due to the variation in the thermal processing modes of the material with the UFG structure, microstructures with given grain sizes of the  $\gamma$ -phase (from 10 to 67  $\mu\text{m}$ ) and coherent precipitates of the hardening  $\gamma'$ -phase can be formed, which will make it possible to achieve the required heat-resistant properties.

It is shown that additional thermomechanical treatment in the two-phase region of the EP741NP alloy with the UFG structure allows obtaining a regulated change in the microstructure along the radius of a GTE disk part (UFG microduplex structure in the hub, a “necklace” structure in the web and coarse-grained with tortuous grain boundaries in the rim) and the required functional-gradient properties.

In this paper, heat resistance characteristics (time to failure at a given stress ( $\sigma = 980$  MPa), temperature ( $t = 650$  °C) were investigated in all zones of the disk under the same conditions, which makes it difficult to evaluate the effectiveness of forming a gradient structure for obtaining functional-gradient properties in the GTE disk from the EP741NP powder alloy. The results of research into the impact of a gradient structure on characteristics of the ultimate strength limit in various zones of the model disk made from the EP741NP powder alloy will be presented by the authors in the next paper.

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