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# The Influence of Macrostructure of Nickel-based Superalloys IN713C and MAR 247 on the Characteristics of High-temperature Creep

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## Abstract

The study consisted in assessing the influence of surface and volume modification on the characteristics of high-temperature creep of castings made of waste products of nickel-based superalloys IN 713C and the MAR-247. The results of high-temperature creep tests performed under conditions of two variants of research were analysed. The characteristics of creep according to variant I were obtained on the basis of earlier studies of these alloys with the parameters  $T=982^{\circ}\text{C}$ ,  $\sigma=150\text{MPa}$  [1]. Variant II included carrying out creep tests of alloy IN713C with the parameters  $T=760^{\circ}\text{C}$ ,  $\sigma=400\text{MPa}$  and alloy MAR247 with the parameters:  $T=982^{\circ}\text{C}$ ,  $\sigma=200\text{MPa}$ . Developed creep characteristics were compared with the results of these alloys with the parameters according to variant I of the study. It was observed that the conditions of experiments carried out depending upon the value of the creep test temperature and stress with the creep stability depends on the size of the macrograin (I variant of the studies) or such influence was not observed (II variant of the studies). Stability of samples with coarse structure in variant I of creep tests was significantly higher than the samples with fragmented grain. It was found that the observed stability conditions are dependent on the dominant deformation mechanisms under creep tests carried out - diffusion mechanism in variant I and a dislocation mechanism in variant II of the study. The conditions for the formation and growth of the cracks in the tested materials, including the morphological characteristics of their macro- and microstructure were tested.

**Keywords:** Nickel-base superalloy IN-713C and Mar-247, Macrostructure, Modification, Creep

## 1. Introduction

One of the main problems in the technology of casting heat resistant nickel alloys is the ability to shape their structure, providing the desired casting properties. Combination of creep strength, endurance under thermo - mechanical fatigue and plasticity of nickel-based superalloys, optimized for specific applications in aviation gas turbines can be achieved for example

by a suitable selection of size, orientation and uniformity of grains.

Creep-rupture strength of nickel-based superalloys increases along with the grain size under certain conditions of heat and mechanical loads. In many cases, however yield strength and tensile strength decreases. Moreover, fine-grained structure of castings are characterized by a higher speed of steady creep [1-4].

Therefore, it is reasonable to master the ability to control the shaping of the original structure of these alloys, adequate to the working conditions of the items produced. Methods for controlling grain size of the cast heat-resistant nickel alloys include mainly; modification of the surface, subjecting a solidifying alloy to mechanical factors and volumetric modification of a liquid alloy by introducing appropriate additives, heterogeneous nucleants [5-7]. In the literature, there is a lot of information about the microstructure refinement of nickel superalloys using a refining method [8] and modifying with micro additives [9-10].

The study consisted in assessing the influence of surface and volume modification and double filtration during pouring the moulds on the stability under accelerated creep of castings made of waste products of nickel-based superalloys IN-713C and the MAR-247. The influence of the size of micrograin on the characteristics of high-temperature creep under the conditions of two variants of the study was analysed. Creep characteristics of variant I were obtained on the basis of previous studies of these alloys with the parameters  $T=982^{\circ}\text{C}$ ,  $\sigma=150\text{MPa}$  [1]. Variant II included carrying out alloy creep tests of IN713C with the parameters  $T=760^{\circ}\text{C}$ ,  $\sigma=400\text{MPa}$  and alloy MAR247 with the parameters  $T=982^{\circ}\text{C}$ ,  $\sigma=200\text{MPa}$ . The studies simulated destruction processes observed in the extreme conditions most strenuous parts of turbine engines. Conditions for the formation and growth of the cracks in the samples, taking into account the stereological characteristics of macro- and microstructure of materials, were analysed. Results of laboratory tests allow an initial assessment of the suitability of different technologies of modifying nickel-based superalloys for specific applications in aviation gas turbines.

## 2. Material and research methodology

Four groups of threaded samples with dimensions (M12,  $d_o=6,0\text{ mm}$ ,  $l_o=32\text{mm}$ ) were prepared. Samples for mechanical testing of macro- and microstructure were made of castings for which a starting batch material was waste from nickel-based superalloy IN-713C and the MAR-247 with the chemical composition showed in the Table 1.

Table 1.

The chemical composition of nickel-based superalloys

Material	Content, % by mass					
	Cr	Mo	Co	Ti	Al	Nb
IN-713C	13	5.76	0.045	0.99	5.91	3.01
	W	C	Hf	Ta	Si	Zr
	0.04	0.085	0.02	0.05	0.006	0.06
	Ni – 71.5					
MAR-247	9.05	0.78	9.48	1.12	5.52	0.12
	W	C	Hf	Ta	Si	Zr
	11.2	0.112	1.18	3.78	0.08	0.04
	Ni – 57.3					

Castings were obtained as a result of the following four casting experiments:

1. Cast IN-713C, (a form of blue, blue filter)

2. Cast IN-713C, (a form of white, blue filter)

3. Cast MAR-247, (a form of blue, blue filter)

4. Cast MAR-247, (a form of white, blue filter)

The process of smelting waste products in the crucible of  $\text{Al}_2\text{O}_3$ , and then casting was conducted in a vacuum induction furnace type IS 5/III, by Leybold-Heraeus. Experiment 2 and 4 included shaping a macrostructure of materials solely under the procedure of volume modification. While experiment 1 and 3 under the conditions of combined modification, so called surface and volume modification.

Total procedure of surface and volume modification required the application of so-called "blue" form (with a modifying coating- $\text{CoAl}_2\text{O}_4$ ) and placing additional filter containing also a cobalt aluminate in the runner dish. An additional result of the solution was a double filtration of the alloy.

Creep tests were conducted under conditions corresponding to the operating conditions of turbine blades of aircraft engines. Different test parameters were used including the parameters corresponding to the acceptance tests used in WSK Rzeszów compatible with Technological report cards for a given superalloy (for IN713C:  $T=982^{\circ}\text{C}$ ,  $\sigma=150\text{MPa}$ , for Mar247:  $T=982^{\circ}\text{C}$ ,  $\sigma=200\text{MPa}$ ). Creep tests were performed on the machine Walter-Bai AG LFMZ-30kN. The study was conducted with the parameters listed in Table 1. Table 1 lists also the parameters which determine the deformation mechanisms of this group of alloys (Fig. 1), [11, 12], so called normalized stress  $\tau_N=\tau/G$  and homologous temperature  $T_H=T/T_{top}$ . Variation of creep test parameters (Table 1) was designed to obtain information about the influence of grain size on the creep characteristics of the material. The knowledge in this field will allow shaping a macrostructure of cast components in the foundry processes in a rational way, depending on the applications in aviation turbine engine. Important determinants of the characteristics of the casting macrostructure are primarily the conditions of their thermo-mechanical loads.

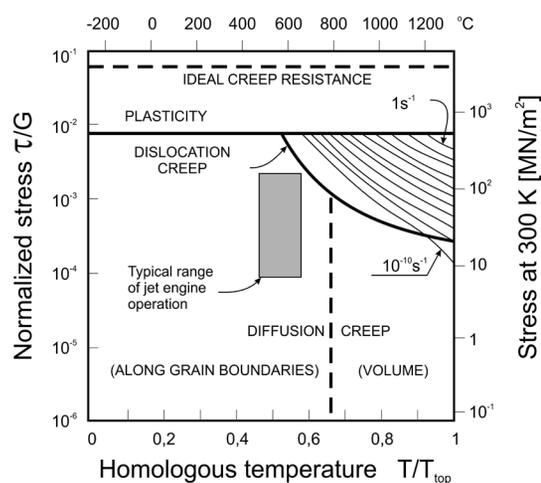


Fig. 1. Map of deformation mechanism of Mar-M200 alloy, grain size 10mm [11]

Table 2.

Parameters of creep tests of nickel -based superalloys IN713C and MAR247

Material	Variant I	Variant II	
IN-713C	Temperature T=982°C	Temperature T=760°C	$T_H = 0.76$
	Stress $\sigma=150$ MPa	Stress $\tau_N \approx \sigma=400$ MPa	$0.0026$
	Homologous temperature $T_H = T/T_{top}$		
MAR-247	$T_{H(IN713C)} \approx 0.76$	Temperature T=982°C	$T_H = 0.77$
	$T_{H(MAR247)} \approx 0.77$	Stress $\tau_N \approx \sigma=200$ MPa	$0.0013$
	Normalized stress $\tau_N = \tau/G \approx 0,001$		

Analysis of the map of deformation mechanisms indicates that plastic deformation in the process of superalloy creep may occur as a result of diffusion or dislocation creep depending on the test conditions (temperature and stress). In the conditions of diffusion creep according to a model of R. L. Coble and Nabarro-Herring steady creep rate significantly depends on the grain size and is described with the relations (1) and (2), respectively [12-14]:

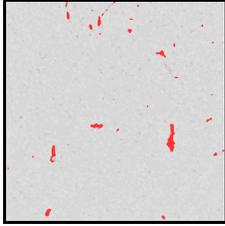
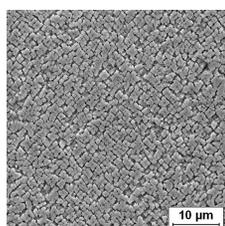
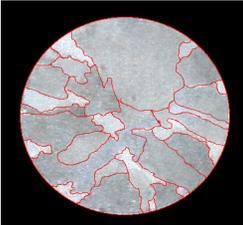
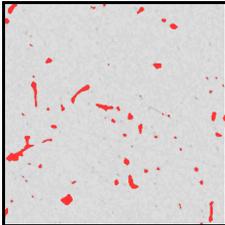
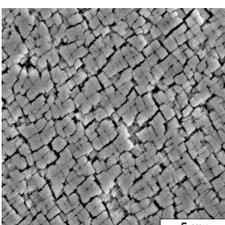
$$\dot{\epsilon} = \frac{B\sigma\delta}{kT} \frac{\Omega D_{GB}}{d^3} \quad (1)$$

$$\dot{\epsilon} = \frac{C\sigma\Omega}{kT} \frac{D_v}{d^2} \quad (2)$$

where: B, C – material constants,  $\sigma$  – stress,  $D_{gz}$  – diffusion coefficient across the grain boundaries, b – the Burgers vector, k – Boltzmann constant, T – absolute temperature, d – grain diameter,  $\Omega$  – atomic volume,  $\delta$  – effective thickness,  $D_v$  – Lattice diffusion coefficient

Table 3.

Summary macro- and microstructure images and its morphological parameters. II variant of the studies

Material	Evaluation of structure		
	Macrostructure parameters	Carbide content in surface area $A_A$	phase form $\gamma'$
Inconel IN-713C - sample 1a fine grain	 N=24, $A=1,11$ mm <sup>2</sup>	 $A_A=1,43\%$	 10 $\mu$ m
Mar M-247 sample 3a fine grain	 N=29, $A=0,90$ mm <sup>2</sup>	 $A_A=2,51\%$	 5 $\mu$ m

N - Number of grains on specimen cross-section, A- Average surface of flat grain cross section

While in case of case of dislocation creep mechanism it is described by the relation (3) and is not dependent on the grain size:

$$\dot{\epsilon} = \frac{AD_{ef}Gb}{kT} \left( \frac{\tau}{G} \right)^n \quad (3)$$

where: A, n – material constants  $\tau$  – shear stress,  $D_{ef}$ -diffusion coefficient, G – shear modulus b – the Burgers vector, k – Boltzmann constant, T – absolute temperature, d – grain diameter.

It should be noted at the same time, that under conditions of creep tests deformation of the material as a result of dislocation creep, volume diffusion (Nabarro-Herring model) and across the grain boundaries (Coble' model) may take place simultaneously with different intensity. The contribution of each of these processes in the deformation depends on the temperature, stress, grain size and the structure of their boundaries [12-13].

### 3. The results of investigations and discussion of results

Images of selected cast structures studied under the conditions of variant II of the creep tests are presented in Table. 3. Preparations for microscopic observation were pickled in the Marble's reagent. Table 4 and 5 list selected morphological parameters of macro- and microstructures of the test samples. Basic parameters of the macrostructure were evaluated using Met-Ilo program. The tests were performed on cross-sections of samples ( $d_0 = 6$ mm) after the creep test.

Table 4.

The parameters of examined nickel-based superalloys structure and creep characteristics determined on samples subjected to creep under the conditions of the I variant

Sample no.	Material	Characteristics of the material	Number of grains on specimen cross-section $N$	Average surface of flat grain cross section $A_s$ , $\text{mm}^2$	Carbide content in surface area $A_A$ , %	Ratio of carbide content in surface area and number of grains $A_A/N$ , %	Steady-state creep rate $V_u$ , %/h	Resistance, time to specimen failure $t_z$ , h
1	IN-713C	fine grain	25	0.86	0.95	0.038	$2.8 \times 10^{-7}$	28
2		large grain	10	2.68	0.86	0.086	$1.38 \times 10^{-7}$	50
3	MAR-247	fine grain	45	0.57	2.12	0.047	$2.5 \times 10^{-8}$	250
4		large grain	7	3.55	1.45	0.21	$2.2 \times 10^{-8}$	317

Table 5.

The parameters of the structure creep characteristics of examined nickel-based superalloys determined on samples subjected to creep under the conditions of the II variant

Sample no.	Material	Characteristics of the material	Number of grains on specimen cross-section $N$	Average surface of flat grain cross section $A_s$ , $\text{mm}^2$	Carbide content in surface area $A_A$ , %	Ratio of carbide content in surface area and number of grains $A_A/N$ , %	Steady-state creep rate $V_u$ , %/h	Resistance, time to specimen failure $t_z$ , h
1a	IN-713C	Small grain	24	1.11	1.43	0.06	$2.0 \times 10^{-6}$	1228
2a		Large grain	13	2.08	1.50	0.115	$2.39 \times 10^{-6}$	1226
3a	MAR-247	Small grain	29	0.9	2.51	0.087	$2.34 \times 10^{-5}$	69
4a		Large grain	7	3.81	2.29	0.327	$2.58 \times 10^{-5}$	65

Metallographic studies indicate that the effect of only volume modification was the formation of coarse-grained structure in superalloys, and simultaneous volume and surface modification resulted in the formation of fine-grained structure (Table 4 and 5). Studies on precipitations of carbide phases, significant from the point of view of strengthening the tested alloys and sustainability in creep conditions showed their greater surface  $A_A$  in superalloy MAR-247 (Table 4 and 5). Primary carbides, mainly in the form of "Chinese characters" occurred in the area of grain boundaries [2].

Tab. 4 and Table 5 summarizes macrostructure stereological parameters of examined superalloys in relation to the creep characteristics such as sample rupture time  $t_z$ , steady creep speed  $V_u$ . These values are important in defining the factors that determine the stability of materials under high-temperature creep.

Figure 2 and 3 shows characteristics of creep of superalloys IN-713C and MAR-247 developed on the basis of creep tests carried out in accordance with variant I of the study.

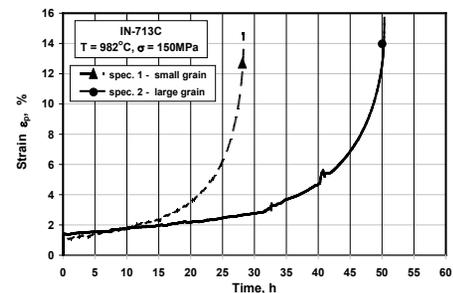


Fig. 2. Creep characteristics of nickel superalloy IN-713C under the conditions of the I variant

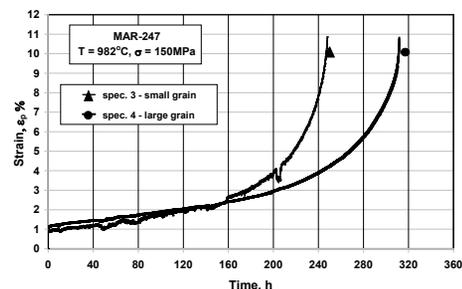


Fig. 3. Creep characteristics of nickel superalloy MAR-247 under the conditions of the I variant

In case of superalloy IN-713C stability will substantially depend on the size of the macrograin and reaches the value  $t = 50$  hours for a sample with a coarse-grained structure and 28 hours for the sample with the comminuted grain as a result of the volume and surface modification (Table 4). Similarly, in a high-temperature creep of alloy MAR-247 the size of the macrograin fundamentally influences samples rupture time. Stability of the samples with a coarse-grained structure was over 20% greater than the comminuted grain samples.

As is clear from the data presented in Table 4 stability of the materials tested was furthermore strongly dependent on the the area of  $A_A$  carbides disclosed in their microstructure. This effect is well illustrated by new parameter  $A_A/N_s$  (surface area of carbides referred to the number of grains in the sample table, Table 6). Regardless of the tested superalloy with an increase in this parameter stability in the creep test  $t_r$  was higher, and the steady creep speed  $V_u$ , reached lower values (Table 4).

The results of research and analysis indicate that diffusion creep across grain boundaries determined the steady creep speed  $V_u$ , and stability of superalloys in completed tests (Table 4). We can assume that in the given circumstances of the I test variant ( $t = 980^\circ\text{C}$ ,  $\sigma = 150\text{MPa}$ ) stability (time to sample rupture) under diffusion creep determined the slip across the grain boundaries. It conditioned the processes of formation and growth of cracks. In this case the decisive factor for the stability of the superalloy was the ratio of the surface area of the carbides to the amount grains on the cross-section of the sample ( $A_A/N$ ). Higher value of this expression corresponds to greater stability of the material in a creep test.

The analysis of the test results obtained with the parameters corresponding to variant II of creep tests (Fig. 4, 5, Tab. 5) indicates that, by increasing the axial stress  $\sigma$  (which results in the increase of normalized stress  $\tau/G$ ) no influence of the macrograin size on the creep stability was observed both in case of superalloy IN-173C and MAR-247 (Fig. 4 and 5). Differences in creep durability were only few hours. This shows that under these creep test conditions the material deformation process takes place mainly under dislocation mechanism, rather than, as previously observed (Fig. 2, 3) under Nabarro-Herring matrix diffusion mechanism (volume) and across the grain boundary by Coble (this resulted in the increase in the stability of the material with a coarse-grained structure). Described influence of creep test parameters on change of materials deformation (distortion) mechanisms due to the increase of the axial stress  $\sigma$  is well explained by figure 6.

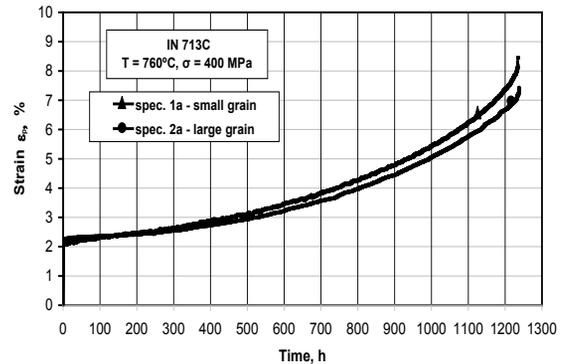


Fig. 4. Creep characteristics of IN-713C nickel superalloy under the conditions of the II variant

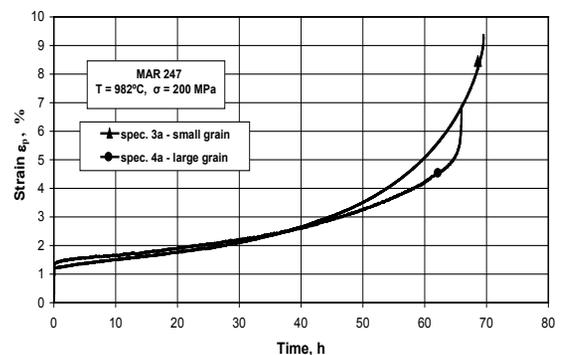


Fig. 5. Creep characteristics of MAR-247 superalloy under the conditions of the II variant

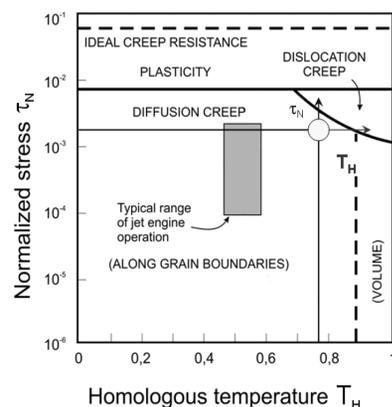


Fig. 6. A map deformation mechanisms of superalloy MAR-200 for a grain size of 0.1 mm (according to M. F. Ashby [13]). A figure schematically indicated what is the direction of the influence of increased normalized stress  $\tau_N = \tau/G$  and homologous temperature ( $T_H = T/T_{top}$ ) in the creep test on the change of creep mechanism from a diffusion creep to dislocation one

## 4. Conclusion

In summary, it can be concluded that under the conditions of creep tests conducted according to the I variant, a crucial factor determining the increase of stability of superalloys with a bigger macrograin (Fig.3, 4) was levelled through an increase in grain size debilitating the actions of grain boundaries. At high temperatures, the grain boundaries absorb dislocations moving as a result of slipping and climbing, which leads to the reduction of strain hardening and weakening of the material. Similarly, weakening effect of grain boundary is associated with the deformation of the material due to the so-called slip across the grain boundaries facilitated in high temperature. The effect is that the grains can slide relative to each other rigidly without appreciable distortion inside the grains. As a result of these processes under these test conditions, a reduced stability and greater deformation rate of the fine-grained material is observed (Fig.3, 4).

Whereas, under the conditions of variant II of the creep test as a result of the increase in value of the normalized stress  $\tau_N = \tau/G$  a factor fundamentally conditioning the deformation and stability of the superalloys (Fig. 5, 6) is no longer a grain size (as can be inferred from the analysis of the deformation map). Under such thermal and mechanical loads a material deforming process takes place in the entire volume of the material, mainly due to a dislocation mechanism (due to the climb and dislocation slip). The role of the grain boundaries in this case is secondary. This is confirmed by the results of creep tests, which showed comparable stability of the samples both with smaller and bigger macrograin (Fig. 5, 6).

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