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## IMPACT OF A STRUCTURE ON DURABILITY OF MODIFIED NICKEL-BASE SUPERALLOYS IN CREEP CONDITIONS

### WPLYW STRUKTURY NA TRWAŁOŚĆ W WARUNKACH PEŁZANIA MODYFIKOWANYCH NADSTOPÓW NA BAZIE NIKLU

The study assesses the impact of surface and bulk modification and filtration during pouring on a durability under accelerated creep conditions of casts made of IN-713C and MAR-247 nickel superalloys scrap used for manufacturing of aircraft engine parts. The impact of solutionizing (1185°C/2 h) with subsequent ageing (870°C/20 h) on the creep resistance of a casting made from MAR-247 coarse-grained superalloy was also examined. Morphological structure parameters were determined with the use of Met-Ilo software. Macrostructure analysis of casts showed very significant impact of surface modification treatment. Creep test results clearly showed that coarse grained samples of IN-713C and MAR-247 superalloys have higher creep resistance. Moreover alloy MAR-247 had higher creep durability after heat treatment compared to as-cast state.

*Keywords:* nickel alloys, creep, macrostructure, microstructure, modification

W pracy przeprowadzono ocenę wpływu zabiegów modyfikacji powierzchniowej i objętościowej oraz filtracji podczas zalewania form na trwałość w warunkach przyspieszonego pełzania odlewów wykonanych z odpadów poprodukcyjnych nadstopów niklu IN-713C i MAR-247 stosowanych na łopatki turbin silników lotniczych. Analizie poddano również wpływ operacji przesycań (1185°C/20 h) z następnym starzeniem (870°C/20 h) na trwałość pełzaniową odlewu wytworzonego z stopu MAR-247 o strukturze gruboziarnistej. Badania makrostruktury wykonanych odlewów wykazały bardzo istotny wpływ zabiegu modyfikowania powierzchniowego. Stosując program Met-Ilo określono istotne z punktu widzenia trwałości parametry morfologiczne struktury. Próby pełzania wskazuje, że nadstopy IN-713C i MAR-247 charakteryzujące się większym makroziarnem wykazywały dłuższy czas do zerwania próbki. Ponadto próbki stopu MAR-247 poddane obróbce cieplnej wykazują większą odporność na pełzanie niż próbki w stanie lanym.

### 1. Introduction

The efficient application of heat resistant nickel superalloys used in turbine blades of aircraft engines requires an understanding of the creep mechanisms dominant under certain operating conditions [1-5]. In case of cast nickel superalloys it is particularly important to assess the impact of the chemical composition, casting conditions and grain size modification processes, all of which determine the morphology of macro- and microstructure, on the stability in the conditions of creep tests [6-9] for these tests provide information about the behaviour of material in extreme working conditions.

Different mechanisms of creep are often observed during the short and long-term creep tests [8-11]. In general, the dislocation creep dominate in the short-term trials, whereas diffusion creep (Nabarro-Herring and Coble creep) usually dominate in the long-term studies [10-15]. The diffusion along the grain boundaries proceeds more rapidly than the grain volume diffusion, due to the considerably larger value of the activation energy for the latter [13]. Models of diffusion creep describe the high-temperature creep by significant approxi-

mation, because they don't take into account the dislocation creep mechanism [14]. Understanding of creep mechanism prevalent in certain conditions of temperature and strain is necessary for rational selection of the cast nickel superalloys for specific applications. This knowledge will enable proper selection of chemical composition and modification processes for shaping optimal macro- and microstructure of the castings. Deformation mechanism maps (Fig. 1) are particularly useful in this regard. Using this maps it is possible to predict steady creep rate in specific working conditions and, by extension, anticipate the durability of parts.

The main group of alloys that meet the quality requirements for most demanding parts of aircraft engine turbine (i.e. turbine disks, blades and nozzles) are modern cast cobalt and nickel superalloys. This group include alloys such as: IN-100, IN-738, IN-713C, RENE-77, RENE-80, MAR-257 and MAR-509. These are precipitation-hardened alloys which develop specific macrostructure during solidification consisting of equiaxial and columnar grains and frozen crystals zone. Such structure can be the cause of formation and propagation of cracks and thus cause serious accidents. One of the

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methods of structure improvement is surface [16] and volume [8,9,17,18] modification.

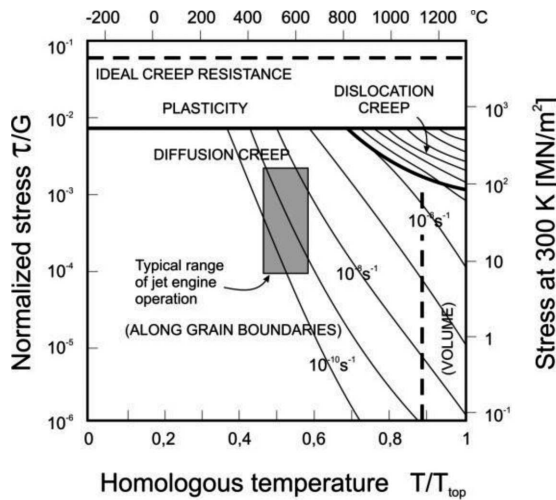


Fig. 1. Map of deformation mechanism of MAR-M-200 alloy, grain size  $100 \mu\text{m}$  [12]

This study assessed the effect of surface and volume modification and double filtration during pouring on the stability under accelerated creep of castings made from scrap IN-713C and MAR-247 nickel superalloys. The study simulated the process of destruction observed in the most extreme working conditions of aircraft turbine parts. Conditions of crack formation and propagation with reference to morphological characteristics of macro- and microstructure were studied. Laboratory findings provide an initial assessment of the suitability of different modification methods of nickel superalloys for specific applications in turbojet engines.

## 2. Methods and materials

Samples for micro- and macrostructure observation and creep tests were taken from castings made from scrap of IN-713C and MAR-247 superalloys. Grain size modification was realised by using  $\text{CoO}\cdot\text{Al}_2\text{O}_3$  coatings applied on mould internal surface and on the ceramic filters commonly used for liquid metal filtration in casting processes. Castings were produced in four following experiments:

1. IN-713C cast to modifying (“blue”) mould with non-modifying (“white”) filter
2. IN-713C cast to non-modifying mould with non-modifying filter
3. MAR-247 cast to modifying mould with modifying filter
4. MAR-247 cast to non-modifying mould with modifying filter

Melting and casting of superalloys scrap was performed in Leybold-Heraeus JS5/III vacuum induction furnace in  $\text{Al}_2\text{O}_3$  crucible. In the experiment 2, macrostructure of material was formed in conditions of volume modification treatment only, while in the experiment 1 and 3 in combined modification treatment (surface and volume modification) experiment 4 delivered reference material for test results from experiment 2.

Combined surface and volume modification requires the use of so called “blue” mould (with a modifying coating of  $\text{CoO}\cdot\text{Al}_2\text{O}_3$ ) with additional modifying filter with  $\text{CoO}\cdot\text{Al}_2\text{O}_3$

coating in pouring basin ( Fig. 2). Additional effect of this method was double alloy filtration [9]. In addition parts of casting made in experiment 4 were subjected to precipitation hardening heat treatment ( solutionizing at  $1185^\circ\text{C}/2 \text{ h}$  and aging at  $870^\circ\text{C}/20 \text{ h}$ ).

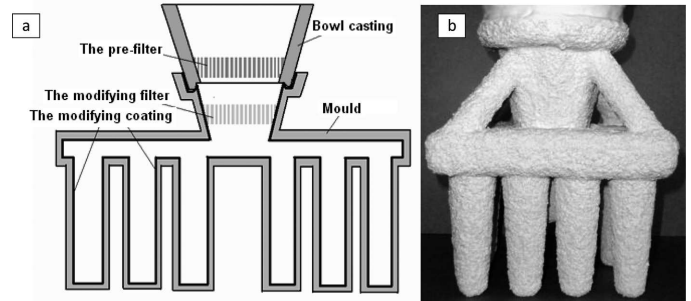


Fig. 2. Schematic of the ceramic mould (a), assembled mould (b)

Threaded samples (M12;  $d_0 = 6,0 \text{ mm}$ ;  $l_0 = 32 \text{ mm}$ ) were made for creep tests (Fig. 3). Creep tests were performed in Walter-Bai AG LFMZ-30kN testing machine at the temperature of  $982^\circ\text{C}$  and axial load causing the stress  $\sigma_0 = 150 \text{ MPa}$  in cross-section area of specimens. Creep tests were carried out in simulated conditions corresponding to extreme operating conditions of turbojet engine blades. In the conditions of performed tests normalized stress  $\tau/G$  was approx.  $10^{-3}$ , at homologous temperature  $T/T_{top} \approx 0.78$ , which allows to assume, based on analysis of deformation mechanisms maps for this group of alloys (Fig. 1), that deformation of the analysed superalloys samples will occur mainly as the result of diffusion creep, owing to diffusion of atoms along grain boundaries [6,12,13].

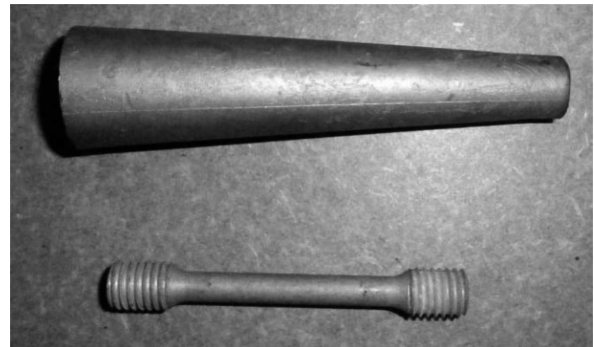


Fig. 3. Cast and sample for creep test

Structure analysis was done on the samples used in creep tests. Microstructure observation were performed using Hitachi 4200 SEM microscope. Specimens for macrostructure observation were made from cross-sections of the creep test samples by etching with Marble solution. Basic parameters of structure were evaluated with the use of Met-Ilo image analysis software.

## 3. Results and discussion

Macrostructure parameters of castings obtained during the study were collated in TABLE 1. Macrostructure analysis of IN-713C and MAR-247 castings, which have the tendency

to form coarse grained structure in selected casting conditions (experiments 2 and 4), shows that surface modification process (experiments 1 and 3) leads to development of fine grained structure (TABLE 1).

TABLE 1  
Stereological parameters of macrograins observed in superalloys  
IN-713C and MAR-247

Experiment	Material	Number of grains, $N^*$	Average grain surface area, $A$ [ $\text{mm}^2$ ]*	Carbide content per surface area $A_A$ [%]**
1	IN713C	25	0,86	0,95
2		10	2,68	0,86
3	MAR-247	45	0,57	2,12
4		7	3,55	1,45
4 after HT		10	2,68	1,91

\* measured on the cross-section of the sample

Microstructure observations indicate the presence of primary carbide phases mainly in the areas of grain boundaries. Larger carbide content per surface area  $A_A$  was observed in MAR-247 samples (TABLE 2). Figs. 4 and 5 show characteristic carbide precipitation arrangement in IN-713C and MAR-247 alloy samples from experiments 2 and 4.

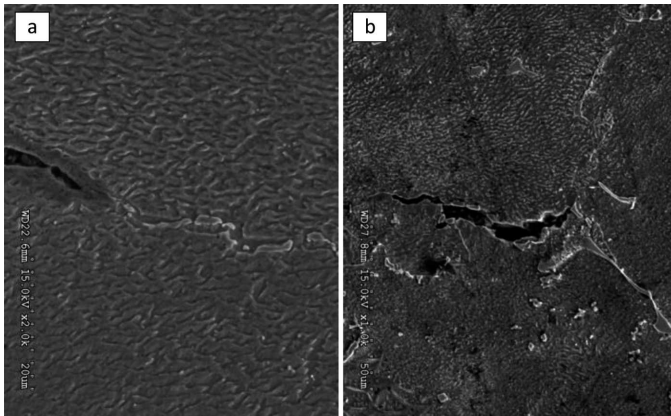


Fig. 4. Effect of crack blocking on precipitations of primary carbides within the grain boundaries, a) IN713C alloy, b) MAR-247 alloy

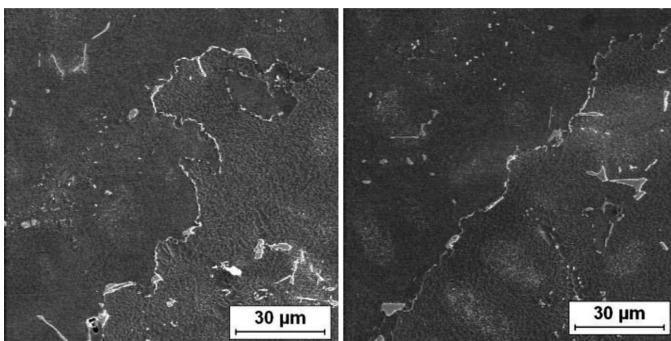


Fig. 5. Microstructure of IN-713C superalloy

Precipitates of  $\gamma'$  phase in as-cast samples were cuboidal with edge length of approximately  $0.4 \mu\text{m}$  ( Fig. 6a). In MAR-247 alloy after precipitation hardening heat treatment primal  $\gamma'$  precipitates were broken to smaller cuboidal par-

ticles with edge length of approximately  $50 \text{ nm}$  (Fig. 6c,d). Fragmentation of primal  $\gamma'$  precipitates proceeded from surface to the core of the particle and was occurring uniformly thorough the volume of the alloy. After the creep tests in all studied cases the morphology of  $\gamma'$  precipitates changed from cuboidal to more oval as a result of coagulation ( Fig. 6b).

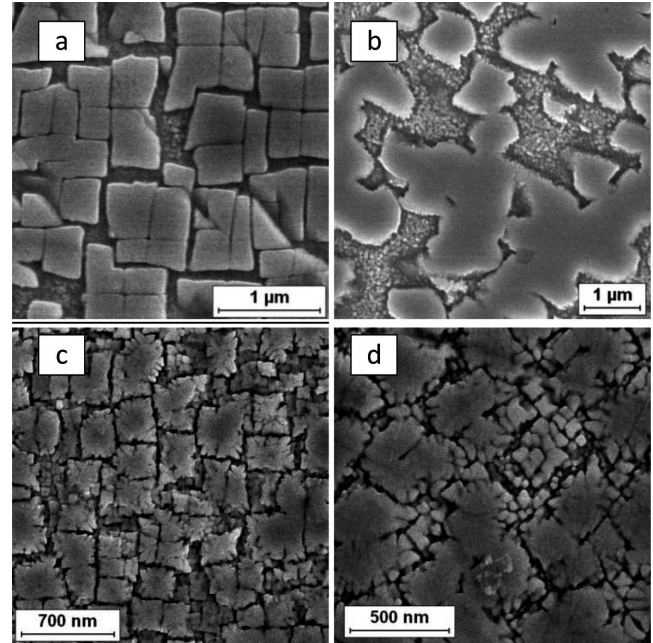


Fig. 6. Morphology of  $\gamma'$  phase in alloy MAR-247. Initial conditions (a), conditions as after creep process (b), condition after heat treatment (c, d)

TABLE 2  
Creep resistance and selected stereological parameters of  
microstructure

Experiment	Creep resistance, $t_f$ [h]*	Steady state creep rate $V_s$ [1/s]	Carbide content per surface area $A_A$ [%]**	Number of grains, $N^{**}$
1	28	$2,8 \times 10^{-7}$	0.95	25
2	50	$1,38 \times 10^{-7}$	0.86	10
3	250	$2,5 \times 10^{-8}$	2.12	45
4	317	$2,2 \times 10^{-8}$	1.45	7
4 after HT	353	$1,06 \times 10^{-8}$	1.91	10

\* measured on the cross-section of the sample

\*\* time to rupture

Fig. 7 and Fig. 8 show creep characteristics of studied nickel-base superalloys. In TABLE 2 morphological parameters of macrostructure were compared with time to failure  $t_f$  and steady state creep rate  $V_s$  which are important for determination of material durability under creep conditions [1,13,19].

Analysis of the obtained results shows that in conditions of high-temperature creep the durability  $t_f$  of coarse-grained IN-713C superalloy is approximately two times higher as compared to sample with grain size reduced in result of surface modification. As shown in TABLE 2, the durability of studied materials was strictly related to content of carbides in surface area of the specimen  $A_A$ . With the increase of this parameter

the durability  $t_f$  rose and the steady creep rate  $V_s$  decreased. This explains well the impact of carbide precipitations on the inhibition of crack growth, i.e. by suppression of grain boundary slide.

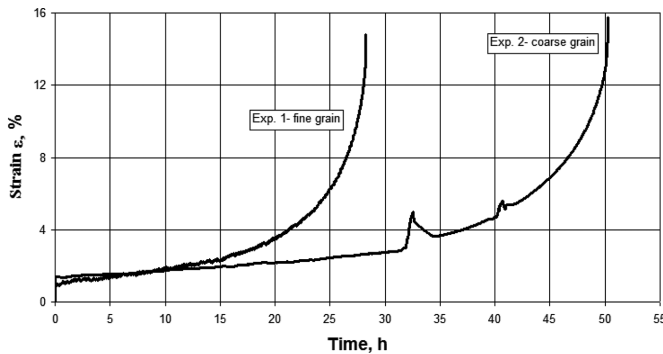


Fig. 7. Creep characteristics of nickel superalloys IN-713C

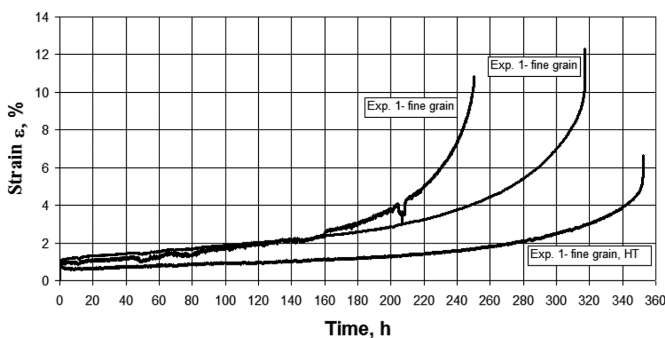


Fig. 8. Creep characteristics of nickel superalloys MAR-247

Analysis of creep test results for MAR-247 alloy shows that for unmodified, coarse grained structure the creep durability was about 27% compared to fine grained sample. Moreover after heat treatment of samples from experiment 4 the creep durability increased by additional 11% (TABLE 2).

Increased durability after heat treatment of MAR-247 alloy is associated with the refinement of  $\gamma'$  particles. Fine particles contribute more to solution strengthening by creating additional obstacles for dislocation movement. This results in blocking the slides responsible for shape deformation in high temperature and increase of MAR-247 durability observed in the study.

An equally important information resulting from the creep test ( Fig. 8) is that the MAR-247 alloy subjected to heat treatment was characterized by smaller deformation to rupture when compared to the initial, as cast state. However, under the conditions of creep, the heat-treated alloy showed smaller steady-state creep rate and, at the same time, longer time of the stage II of creep ( Fig. 8, TABLE 2). This property of the superalloy should be considered very desirable, as it may result in longer life for example of blades used in aircraft jet turbines.

Analysis of characteristic curves of creep for studied superalloys indicates that diffusion creep process by grain boundaries determine their resistance in the conducted creep tests. Coarse grained specimens of IN-713C was characterised by higher creep resistance  $t_z$  with lower stable creep rate  $V_u$  (TABLE 2). Analysis of cracking processes shows that, in given

conditions ( $T = 982^\circ\text{C}$ ,  $\sigma = 150\text{ MPa}$ ), crack development was determined by grain boundary slip in the conditions of diffusion creep.

#### 4. Conclusion

Analysis clearly shows that coarse grained samples of IN-713C and MAR-247 superalloys have longer time to rupture. This shows that the creep resistance in conditions of this study is determined by grain-boundary diffusion creep. Coarse grained specimens of IN-713C was characterised by higher creep resistance  $t_f$  with lower stable creep rate  $V_s$  (TABLE 2). Moreover alloy MAR-247 had higher creep durability after heat treatment compared to as-cast state.

Higher durability of MAR-247 in comparison to IN-713C results from higher concentration of carbide precipitations in the structure of MAR-247 superalloy (TABLE 2). Durability is also affected by higher concentration of  $\gamma'$  phase, addition of 1,3% Hf to strengthen the  $\gamma'$  phase, addition of B to strengthen the grain boundary and positively influence the morphology of grain-boundary carbide precipitations. Additionally other alloying elements of MAR-247 (Co, W, Mo, Cr, Ta, Hf) strengthen the  $\gamma$  matrix and weaken the diffusion processes contribution to higher creep resistance in comparison to IN-713C alloy.

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