

Zdenek Jonsta – Petr Jonsta – Karel Mazanec *

MICROSTRUCTURAL MATERIAL ANALYSIS OF SUPERALLOY INCONEL 792-5A AFTER HIGH TEMPERATURE EXPOSITION

Aerospace industry often uses nickel superalloys for blades of jet engine turbines. The reason is that this material can satisfy numerous extreme requirements, such e.g. strength even at very high temperatures, resistance to fatigue damage, resistance to fatigue effect of combustion gases, etc. Long-term service life and material reliability is directly linked to its microstructure, or with its stability at long-term exploitation. Presented article deals with analysis microstructural material characteristics of cast variants nickel superalloy of INCONEL 792-5A type. Several strengthening mechanisms are applied in this type of superalloy. Principal mechanism is precipitation strengthening by coherent precipitates of intermetallic phase Ni_3Ti , or $Ni_3(Ti,Al)$. The analysis as such is based on evaluation of microstructural parameters by application of electron microscopy and chemical microanalysis.

1. Introduction

The paper is oriented on structural material analysis of cast nickel superalloy of the INCONEL 792-5A type after long-term annealing at temperature of 900 °C. From the viewpoint of practical exploitation this represents the upper limit of efficient use. Importance of solution consists in the fact that a higher level of alloying this type of superalloy has a higher level of segregation activity. At exploitation this fact can lead to the development of specific microsegregation processes related to a precipitation process and with formation either of carbidic phases, or some variants of intermetallic phases, e.g. of the γ' type - (Ni_3Ti , Al). In our case we applied annealing at the temperature 900 °C for 10^3 and 10^4 hours. For all practical purposes this represents conditions which have undoubtedly response from a technological practice viewpoint and can contribute both to a more objective evaluation of service life of relevant castings and also to the overall evaluation of microstructural stability [6, 8]

2. Experimental equipment

The testing material was after heat treatment to appropriate quality by the mode: 1120 °C/2h/air + 845 °C/24h/air long-term

annealed at temperature of 900 °C for 1000 and 10 000 hours. Chemical composition of investigated material IN 792-5A is given in the following Table I.

For an objective evaluation of stability of this alloy it is necessary to know also the concentration of vacancies \bar{N}_v on a 3d sphere, which is in the given case 2.38. This value represents the level, which will apparently significantly participate in restriction of precipitation of intermetallic σ - phase.

Investigation of properties of evaluated samples was made on a microanalyser JCSA 733, which was equipped with an energy-dispersive analyser EDAX. Evaluation of microstructural characteristics was made in the mode of secondary electrons and bounced electrons (COMPO contrast). Individual coexisting phases were identified by quantitative EDX analysis. It must be, however, noted in this context that semi-quantitative X-ray microanalysis was performed only in case of particles larger than 1 μm , when results are not distorted significantly by X-ray signal from the surrounding matrix.

Microstructure of investigated samples was formed by a γ matrix, in which particles of intermediary phase γ' precipitated [5, 7]. It is known from the existing results of analyses of investi-

Chemical composition of IN 792-5A (wt. %)

Table 1

C	Mn(max)	Si(max)	Cr	Ti	Al	Fe(max)	B		
0.06-0.10	0.15	0.20	12.0-16.0	3.75-4.20	3.15-3.60	0.50	0.010-0.020		
Nb	Ta	Mo	W	Cu(max)	Co(max)	P(max)	S(max)	Zr	Ni
0.50	3.85-4.50	1.65-2.15	3.85-4.50	0.50	8.50-9.50	0.015	0.015	0.01-0.05	rest

* Zdenek Jonsta, Petr Jonsta, Karel Mazanec
VSB-TU Ostrava, Czech Republic, E-mail: zdenek.jonsta@vsb.cz

gated variants of heat treatment of high-alloyed Ni-superalloys that in case of application of two-stage heat treatment a bi-modal size distribution of particles of γ' phase is detected. It was observed during a subsequent long-term exposition at temperature of 900 °C that this type of distribution is gradually eliminated. As for the above mentioned discussion and determination of the limit value \bar{N}_v , it is necessary to take into account the fact that during the long-term exposition a concentration of precipitates is being modified. The consequence is that even at the formal value $\bar{N}_v \approx 2.3$ the value \bar{N}_v can be changed up to the level, for example, 2.5 and 2.6 at a formal constriction of the matrix.

Chemical composition of the γ' phase with its certain variability corresponds to the intermetallics $\text{Ni}_3(\text{Ti}, \text{Al})$, whereas this phase dissolves a certain voluminal portion of Ta. Due to a higher segregation activity of Ni-superalloys it is also necessary to take into account the fact that there is a certain difference in the chemical composition of matrix in the area of dendrites and in interdendritic space. This means that the chemical constitution of intermetallic phase γ' will be changing. It is worthwhile to remind that the preferential existence of $\text{Ni}_3(\text{Ti}, \text{Al})$ is to a certain extent linked to a compressibility of the matrix – a higher concentration of electrons in the 3d sphere and fulfilment of the requirement for low compressibility realisation as it corresponds to the condition for formation of the phase γ' [9]. If the concentration of vacancies is higher due to addition of elements with a lower concentration of electrons it is possible to expect, as a result of development of possible compressibility, a formation of the phase with a higher range of specific volume in comparison with the inter-metallic phase γ' (around 1%), namely formation of another type of inter-metallic phase η , characterised as Ni_3X . This phase can be classified into the category of intermetallics γ'' .

3. Basic microstructural analysis

Development of segregation processes during solidification of investigated nickel superalloy is accompanied by a formation of areas

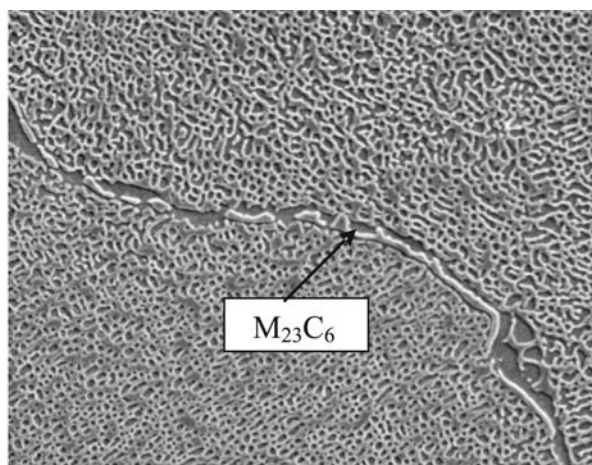


Fig. 1 Precipitation of M_{23}C_6 along grain boundaries surrounded by a film of the phase γ' , magn. 1800x

of a matrix with distinctly different chemical composition, which leads logically to a certain scatter in the finally formed microstructure. In this case a different variant of eutectics is formed in interdendritic areas, which is formed by a mixture of $\gamma + \gamma'$. In the areas of eutectics, which solidified as last ones, coarse particles of γ' were formed. Fine shrinkage porosities were present in some cases in the neighbourhood of eutectic formations. Numerous carbides of the type MX, rich in Ta and Ti, precipitated in interdendritic areas. These particles often decorated the boundaries of γ grains. It is known that carbides of the type MX are unstable in nickel alloys and at higher exposition temperatures they gradually decay, which is accompanied by formation of the particles M_{23}C_6 and the phase γ' [1]. MX particles were in investigated samples “wrapped” by a film of the phase γ' , the thickness of which increases with duration of annealing. Particles of Cr-Mo-W phase or formations of the phase, which is hexagonal phase of the type Ni_3X containing mainly Ti, Ta and Al, were present in proximity of some eutectic formations. Combination of elements in the phase rich in chromium (Cr-Mo-W) is typical for the phase M_6C , however, in the work [2] the particles of similar composition were identified by electron diffraction as borides of the type M_3B_2 , or M_3B_3 .

Occurrence of discontinuous netting of particles of the carbide MX and M_{23}C_6 was detected at the boundaries of γ - grains, which was surrounded by a film formed of the γ' phase. Particles of the phase rich in Cr, or η - phases were observed at the grain boundaries only in very limited quantity. Positive is the fact that occurrence of phases such as σ - phase, μ - phase, R - phase, was not discovered in any variants of evaluated heat treatment [6, 10]. Very important is the fact that at superposed annealing at temperature 900 °C no reprecipitation occurred, consisting in dissolution of the old and formation of the new phase, nor any dissolution of some minority phases. Only processes of growth and coarsening of particles of the phases present in the structure already after duration of annealing of 10^3 hours were running. Apart from the above mentioned facts the particles of M_{23}C_6 after exposition at temperature of 900 °C for 10^4 hours precipitated in the form of incoherent chains in coarse particles γ' in eutectics.

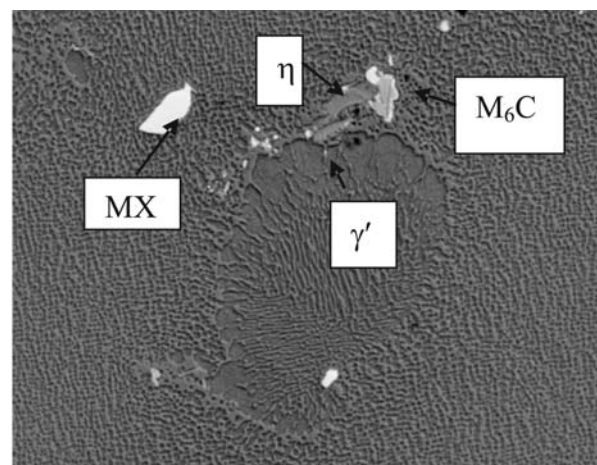


Fig. 2 Eutectics, MX, M_6C , of η phase, SEI, magn. 1700x

Examples of distribution of minority phases along grain boundaries and in proximity of the eutectics $\gamma+\gamma'$ are given in Figs. 1 and 2.

Precipitation is in most cases intergranular or it occurs on the grain boundary $\gamma+\gamma'$, which means a certain risk of initiation of degradation process [3, 4, 11]. Typical EDX spectra of individual minority phases are shown in Figs. 3-8.

4. Microstructural characteristics after various durations of exposition

On the basis of results of microstructural analysis of the investigated nickel superalloy after the relevant exposition durations of isothermal annealing, namely 900 °C/1000 hours and 900 °C/10000

hours, it can be stated that microstructure shows in many cases a specific behaviour. This is manifested by occurrence of various morphologically different variants of minority phases. In the men both in the mode of secondary electrons (SEI), as well as in the mode of bounced electrons (BEI). Two sets of measurement were realised for each variant.

In case of the first variant of heat treatment after 1000 hours of exposition precipitation of γ' - phase on the boundaries of γ - grains or at the phase interface was mostly detected (see Figs. 9 and 10).

Moreover precipitated phases were evaluated by EDX analysis, namely the phases MX, phases rich in Cr, γ' (Ni₃Ti, Al) and chemical composition of matrix was determined. In case of the analysis of chemical constitution of the phase MX a big difference

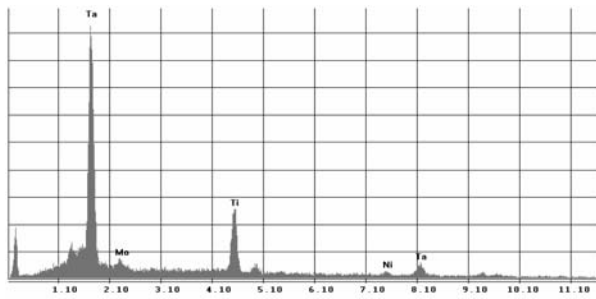


Fig. 3 EDX spectrum of the MX phase

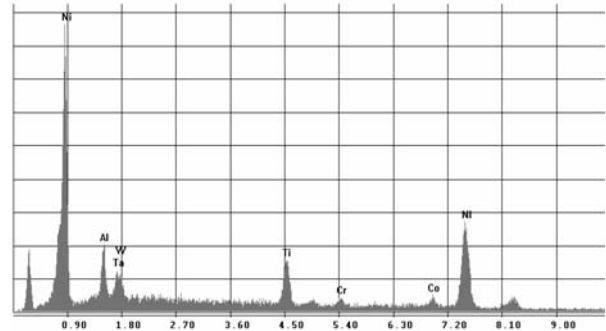


Fig. 4 EDX spectrum of the phase Ni₃(Al,Ti)

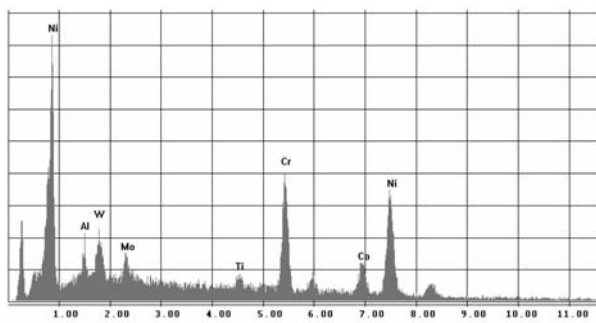


Fig. 5 EDX matrix spectrum

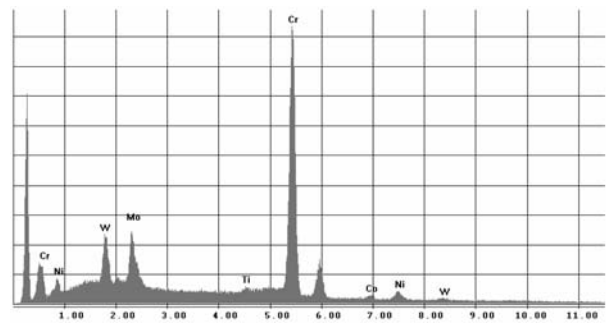


Fig. 6 EDX spectrum of the phase M₂₃C₆

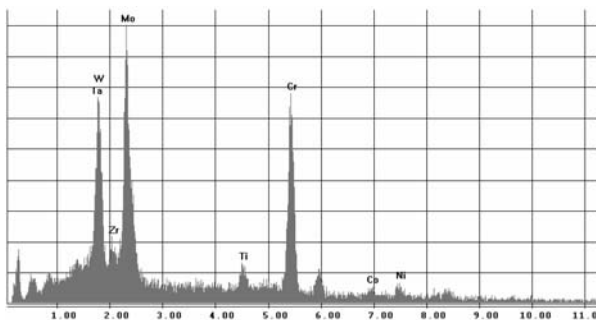


Fig. 7 EDX spectrum of the phase rich in Cr

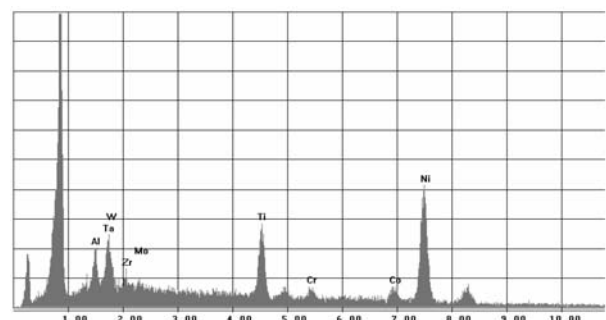


Fig. 8 EDX spectrum of the phase Ni₃Ti

in contents of Ta and Ti was detected. In one case the average contents of Ta was around 75 %, while in the second case it was around 48 %, which is approx. 60 % of the contents determined in the first case. Similar relation was observed also in the case of Ti, which usually acts together with Ta within their coexistence. In the first set of measurements the content of Ti was around 20%, in the second set it was around 45 %. This means that at higher content of Ta the content of Ti was lower. In the second set of measurements the relation of content of these two elements in the phase MX was inverse.

Selected cases of precipitation after the second variant of heat treatment by the mode 900 °C/10000 hours are presented in Figs. 11 and 12.

Special attention was paid also to a chemical analysis - mapping of distribution of individual elements forming the material consti-

tution. In these context distributions of Ni, Ta, Cr, Al, Ti and Mo were investigated. In our opinion intensity of selectively evaluated precipitates slightly increased. The matrix is characterised by a very favourable level of uniform distribution of majority of investigated alloying elements. In connection with solution of microstructural response of the matrix a secondary precipitation was observed of the carbide $M_{23}C_6$ decorating the grain boundaries of the matrix. Results of the MX phase evaluation show a distinct mutual substitutability of Ta and Ti. Reduction of Ta leads to an increase of Ti in this phase and vice versa. It can be generally stated that sum of the content of Ta and Ti gives the overall level of the content of these two elements of approx. 90 %. Chemical composition of the phase $Ni_3(Ti, Al)$ is of a conventional character. It is only appropriate to mention that in case of the lower sum of Ti and Al in this phase a slightly higher content of Ta was observed. In investigated cases the same level of the sum of Ti, Al and Ta was observed, namely approx. 25 %, which can be considered as a certain

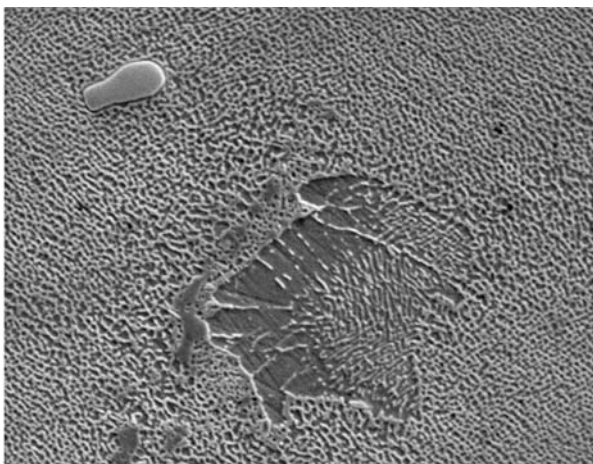


Fig. 9 Precipitation of γ' - phase on the boundaries γ - grains after 1000 hours exposition, SEI, magn. 2200x

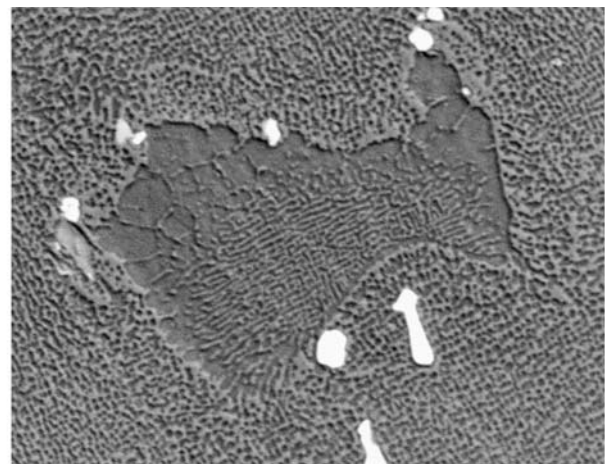


Fig. 10 Precipitation of γ' - phase at the phase interface after 1000 hours exposition, BEI, magn. 1800x



Fig. 11 Precipitation of minority phases after 10000 hours exposition, SEI, magn. 2200x

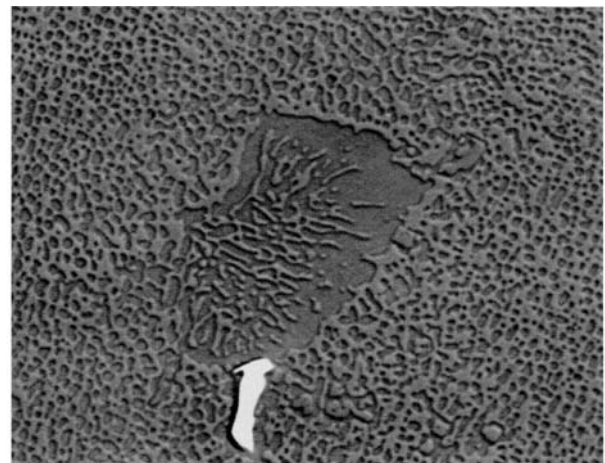


Fig. 12 Precipitation of minority phases after 10000 hours exposition, BEI, magn. 1700x

orientational criterion. The data about chemical composition of the matrix are comparatively surprising, since in one case the content of Ta was determined to be approx. 10 %, while in the second case it was much lower, namely approx. 3.3 – 3.4 %. In this case also a phase rich in Cr was detected, whereas the most important role in chemism of this phase is played by a mutual relation between Cr, W and Mo. Mo content was in both investigated cases detected essentially as constant, specifically around 25–29 %. In case of a higher content of W in this phase the content of Cr was lower, on average around 25 %, while at a lower content of W (around 8 %) the Cr content achieved the value of approx. 40%. As for mutual relation between Ta and Ti, a certain substitutability was discovered in this case too, although obviously at a lower level than in case of MX particles.

5. Conclusion

The paper summarises basic data about structural-phase characteristics of complex alloyed Ni-superalloy INCONEL 792-5A

after high temperature exposition at temperature of 900 °C for 10³ and 10⁴ hours. Apart from the basic microstructural characteristics and monitoring of potential places for formation of a minority phase, an EDX analysis of the possible chemism of precipitated minority phases was performed as well.

Redistribution of atoms of alloying elements in the formed coexisting phases was also analysed. In many instances certain mutual substitutability of alloying elements was observed, which represents an important technical parameter and physical engineering basis for estimation of the type of precipitating phases, as well as for estimation of possible stability of the given Ni-superalloy.

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