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## MICROSTRUCTURAL ANALYSIS OF NICKEL SUPERALLOY MAR-M247

*This paper presents the results of a microstructural phase analysis of the nickel superalloy MAR M247 after casting and different modes of dissolving annealing in the range of 900°C - 1240°C with cooling in water. The alloy in question presents a polycrystalline nickel creep-resistant alloy usable especially for highly stressed components in the aerospace and rocket industries. The analysis was performed using a JEOL JSM 6490LV scanning electron microscope with an Oxford Inca x-act x-ray microanalyzer. The presence of minority phases was identified. The study is focused on their possible changes after the application of different modes of thermal treatment.*

**Keywords:** superalloy, phase analysis, annealing.

### 1. Introduction

The demanding requirements placed on materials working in extreme conditions encourage the use of nickel-based superalloys. The significance of these superalloys for such demanding applications lies primarily in their ability to maintain almost unchanged strength even after long-term exposure to temperatures exceeding 650 °C. One of the most demanding applications is the use of these materials for the hot parts of turbines. The important position of superalloys in this area is reflected in the fact that they currently represent more than 50 % of the mass of modern aircraft engines. The widespread use of superalloys in turbines is supported by the fact that the thermodynamic efficiency of turbines increases with increasing temperatures at the turbine inlet; this has been one factor driving efforts to increase the maximum usable temperature of high-alloyed alloys [1, 2].

The increase in the maximum usable temperature has been enabled particularly by advanced processing techniques which have led to increased purity of alloys and thus to their increased reliability, together with developments in the technique of directional crystallisation and subsequent production technology for products based on single crystals. An equally important factor has been the development of alloys with higher usable temperatures achieved mainly by alloying, especially with Re, W, Ta and Mo [3].

This paper reports on a detailed microstructural phase analysis of the cast nickel superalloy MAR M247 in as-cast condition and after dissolving annealing within the temperature interval of 900 °C - 1240 °C for 2 hours with cooling in water.

The objective of the presented microstructural phase analysis was to achieve a more detailed understanding of this nickel superalloy from the viewpoint of its technical applicability.

### 2. Material and experimental technique

A microstructural phase analysis was performed on the nickel superalloy MAR-M247 in as-cast condition and then after dissolving annealing at temperatures within the interval of 900 °C - 1240 °C. Table 1 gives the chemical composition of the alloy and Table 2 shows modes of dissolving annealing.

The microstructural analysis of the nickel superalloy was performed on the samples in an initial state and after the above-mentioned modes of heat treatment followed by chemical etching. The microstructure was analysed using a light metallographic microscope (Olympus GX51).

Chemical composition in wt. %

Table 1

C	Cr	Mo	Al	Ti	Fe	W	Ta	Zr	Co	Hf	B	Ni
0.16	8.60	0.80	5.60	1.00	0.20	10.00	3.00	0.06	10.00	1.50	0.02	rest

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Modes of dissolving annealing

Table 2

Sample	Temperature / duration of annealing	Cooling
MA VS	as-cast state	water
2MAW	900 °C/2h	water
4MAW	1040 °C/2h	water
13MAW	1200 °C/2h	water
15MAW	1240 °C/2h	water

An electron-microscopic investigation was carried out with a JEOL JSM 6490LV scanning electron microscope equipped with an INCA x-act energy dispersive spectral analyzer. The microstructure was documented in the mode of secondary electrons (SEI) and back-scattered electrons (COMPO - material contrast). Individual phases were identified by high quality X-ray microanalysis. Semiquantitative X ray microanalysis was performed only for the particles bigger than 1 µm, when the results were not distorted significantly by an X-ray signal from the surrounding matrix.

### 3. Results and discussion

The analysed nickel superalloy MAR-M247 is an alloy which is strengthened by the  $\gamma'$  phase in basic austenitic matrix ( $\gamma$ ). The  $\gamma$  matrix in the basic type of this alloy contains approx. 60 vol. % of the phase  $\gamma'$ . Alloying by Co, W, Mo, Ti and other admixtures leads to further substitutive or precipitation strengthening of the matrix and thus to a reinforcing effect of the phase  $\gamma'$ . The addition of C within the interval of 0.05 - 0.2 % leads to the formation of carbides of the type  $M_{23}C_6$ , MC and  $M_6C$  [4]. These carbides are not stable phases. Under the influence of working temperature they may change, including their size and morphology, and by this they influence the properties of the alloy at high-temperature exposure. The microstructure of the as-cast sample (MA VS) was characterised by a typical dendritic character. Segregation processes in

the microstructure during solidification lead to the formation of distinct chemical heterogeneities. Very frequent formations of eutectics  $\gamma + \gamma'$  were segregated in interdendritic spaces - see Fig. 1. These spaces also contain areas that are richer in tungsten. In the areas of eutectics which were the last to solidify, large particles of  $\gamma'$  richer in hafnium were formed. The  $\gamma'$  particles form regular cubes, which are somewhat larger in interdendritic spaces (Fig. 2). Fig. 3 shows a detailed picture of this area.

Particles of primary carbides of the MC type were also identified in the basic matrix which contained variable quantities of carbides of tantalum, titanium, tungsten and hafnium. These carbides are created in the structure as a result of eutectic reaction in the form of larger irregular particles with cubic morphology, and they can be situated randomly both inside the grains and at their boundaries. At high temperature exposure these carbides tend to change into carbides of the type  $M_{23}C_6$  or  $M_6C$ , in case of higher content of molybdenum, tungsten and chromium in the alloy. The influence of a higher content of niobium, which stabilises MC carbides up to the temperature of 1260 °C, plays no role in this case, since this element is not present in the given alloy - see Table 1.

Table 3 below gives chemical composition of identified minority phases.

During solidification, MC carbides are formed into the structure of "Chinese script characters" composed of three different parts [5, 6]. The central part and extended arms contain carbides of the type MC (Ti, Hf, Ta, W) C. The ends of the extended arms are broadened, forming angular "heads". These heads contain carbides of titanium, hafnium, tantalum and tungsten and have an increased content of hafnium in comparison with the central part and the arms. Due to the fact that hafnium strongly segregates to the rest of the solidifying interdendritic melt, interdendritic carbides contain more hafnium than intradendritic carbides [7]. Volume fraction of carbides, their fixation and behaviour during growth are related to their growth rate. Their volume fraction drops a slower growth rate [8], which is caused by their reduced ability of fixa-

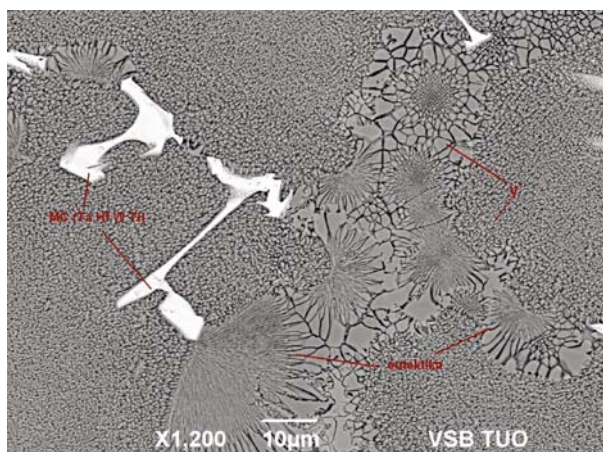


Fig. 1 Microstructure-sample MA VS (BEC)

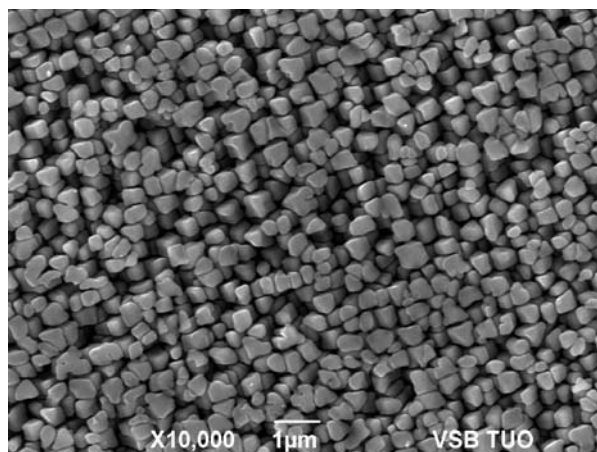


Fig. 2 Phase  $\gamma'$  in matrix (SEI)

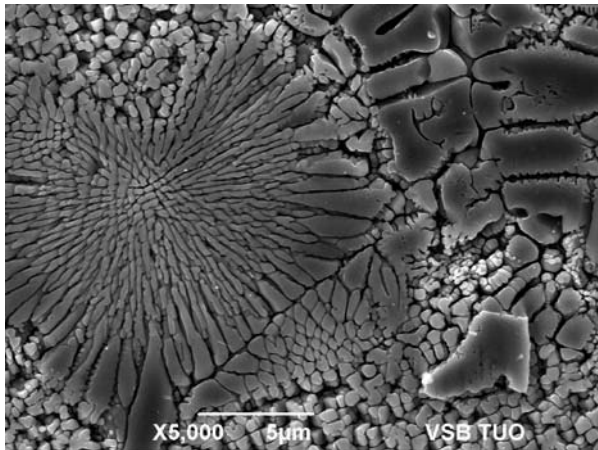


Fig. 3 Formation of eutectics (SEI)

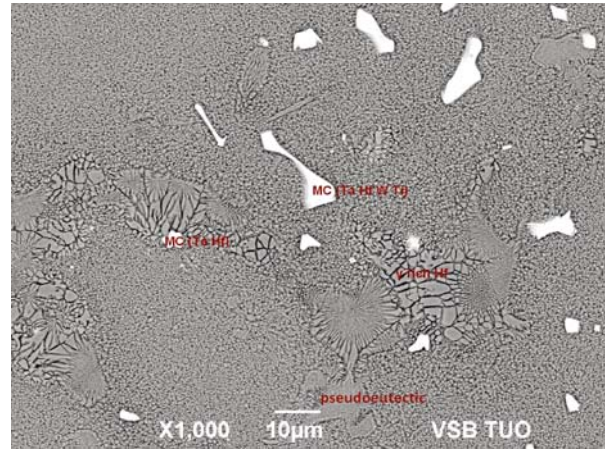


Fig. 4 Microstructure-sample 2MAW (BEC)

Chemical composition of individual phases in wt. % Table 3

Name	Al	Ti	Cr	Co	Ni	Hf	Ta	Mo	W
gamma prime	7.9	2.0	4.2	7.0	64.5	5.5	4.4		4.6
gamma prime Hf-rich	7.1	1.7	3.5	6.4	62.6	10.3	3.8		4.5
eutectics	7.7	1.8	5.6	7.3	64.9	3.3	3.9		5.4
pseudo-eutectics	6.9	1.1	6.2	8.5	60.3	2.2	3.0		11.7
MC (Ta Hf W Ti)		10.3	0.9	0.6	3.6	16.8	53.7		14.1
MC (Hf Ta)		2.7	0.5	0.7	3.9	56.6	32.0		3.6
M <sub>23</sub> C <sub>6</sub>		0.7	36.0	3.3	8.1			9.9	41.9

tion to the solidus - liquidus interface. At slow growth rates the growing carbides become enriched by hafnium and titanium while at rapid growth rates their diffusion is suppressed and carbides contain more tungsten.

After the application of dissolving annealing in the mode of 900 °C/2h/water (sample 2MAW), no important changes occurred in the microstructure as can be seen from Fig. 4 above. During the modes working with higher temperatures of dissolving annealing more significant changes in composition, distribution and morphology of minority phases take place. After dissolving annealing in the mode of 1040 °C/2h/water (sample 4MAW) the hafnium carbides begin to precipitate in the area of eutectics between large particles of  $\gamma'$ . A minority phase rich in Mo, W, Cr, probably carbides M<sub>23</sub>C<sub>6</sub>, is also present. At the same time particles in interdendritic spaces begin to coagulate and to become coarser (Fig. 5).

In the structure of the sample 13MAW treated by the mode 1200 °C/2h/water, it is already possible to see a significant precipitation of carbides rich in hafnium or tantalum.

In the areas of large  $\gamma'$  particles a dissolution of eutectics takes place, as well as a change of size of the  $\gamma'$  particles both inside dendrites and in interdendritic spaces. Bimodal distribution is evident here. Areas containing segregated very fine  $\gamma'$  particles are present in the matrix around the residues of eutectics - Fig. 6.

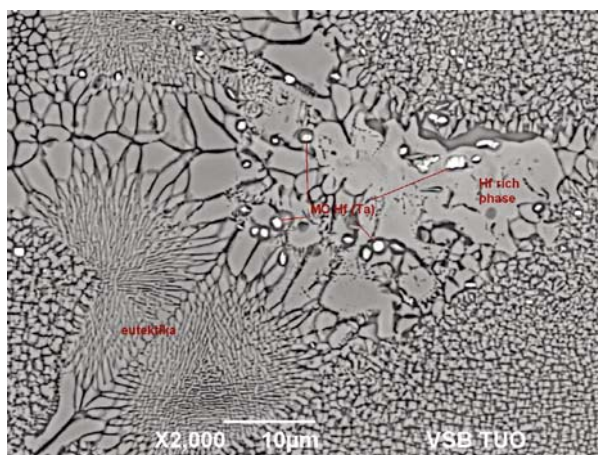


Fig. 5 Microstructure-sample 4MAW (BEC)

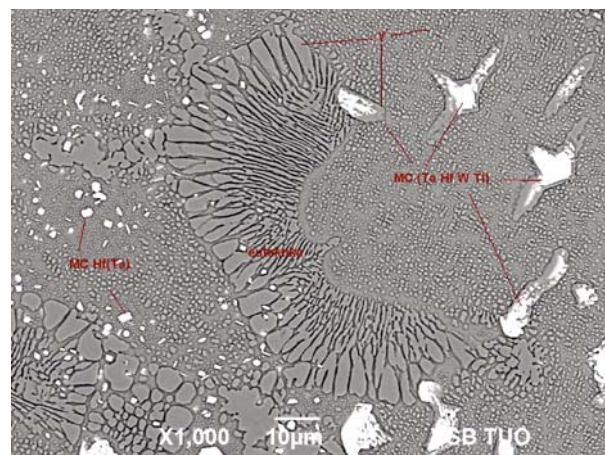


Fig. 6 Microstructure-sample 13MAW (BEC)

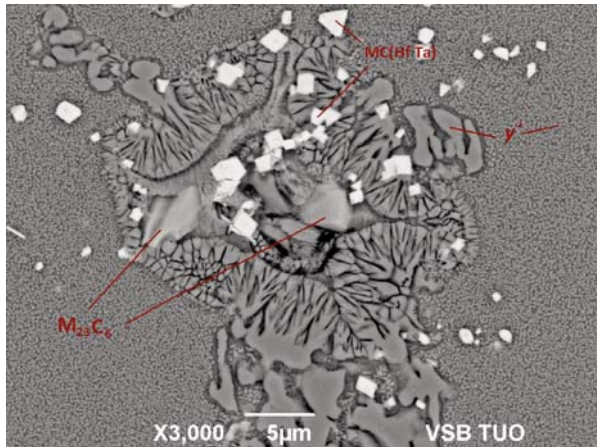


Fig. 7 Microstructure-sample 15MAW - carbides  $M_{23}C_6$  in the centre of residue of eutectic formation (BEC)

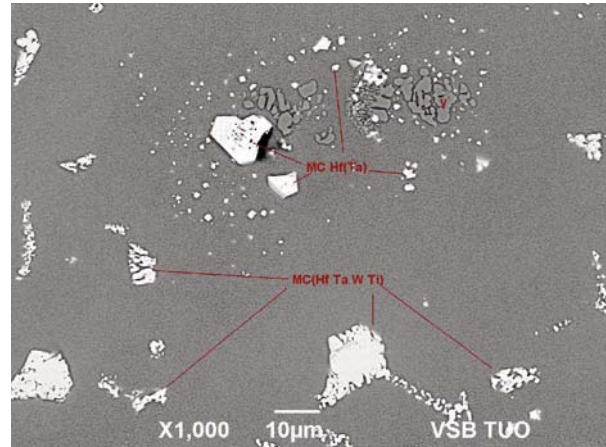


Fig. 8 Microstructure-sample 15MAW - disintegrating primary carbides (BEC)

In the case of the highest temperature of dissolving annealing, namely at the mode of 1240 °C/2h/water in the sample 15MAW, all the  $\gamma'$  particles in the matrix are fine; no bi-modal distribution is observed here. Eutectics are almost dissolved and areas with small carbides of hafnium and tantalum are present at their place. In a few isolated cases residues of eutectics are present, in the centre of which are particles of the carbides  $M_{23}C_6$ .

Large primary carbides disintegrate, and new sharp-edged carbides rich in hafnium and tantalum grow - see Figs. 7 and 8.

#### 4. Conclusions

This paper presented microstructural phase analysis of the nickel superalloy MAR-M247 in as-cast condition and after various modes of dissolving annealing within the temperature interval of 900 - 1240 °C with dwell of 2 hours and cooling in water. It may be stated that changes in the structure of the investigated alloy took place from the temperature of dissolving annealing of 1040 °C.

The increasing temperature of dissolving annealing brought gradual precipitation and increasing frequency of occurrence of hafnium carbides, dissolution of eutectics, and a change of shape

and size of  $\gamma'$  particles both in interdendritic spaces and also inside dendrites. Bimodal distribution was gradually suppressed.

On the basis of X-ray microanalysis (see Table 3) it was possible to observe changes in the percentage representation of individual alloying elements in minority phases.

A quite important fact was that the microstructural phase analysis did not prove the presence of undesirable TCP phases, particularly of the phase  $\sigma$ , which significantly contributes to the degradation of this alloy.

The presented analysis contributes data and serves to deepen knowledge of the investigated alloy for purposes of industrial application.

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