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EFFECT OF THE HEAT TREATMENT ON THE MICROSTRUCTURAL EVOLUTION OF THE NICKEL BASED SUPERALLOY

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Abstract

The effect of heat treatment on the microstructure of cobalt-rich nickel based supperalloy was studied applying scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The aim of the present work was to investigate the formation and evolution of different phases during the heat treatment of superalloy similar to Udimet 500. The presence of a relatively high volume fraction of γ'particles in the γ matrix suggests on inefficient cooling rate during oil quenching from 1150°C. Carbides such as MC primary carbides of the type TiC and MoC as well as secondary carbides $M_{23}C_6$ ($Cr_{23}C_6$) were found in grains and at grain boundaries.
Key words: superalloy; heat treatment;, SEM and TEM; γ' particles; carbides

Introduction

Microstructure of nickel based superalloys highly depends on applied heat treatment. The choice of a heat treatment type has to be adjusted according to required properties such as (hardness, resistance to fracture and corrosion). These properties are mainly the result of influences of two phases. Namely, the γ matrix is FCC solid solution in which particles of the γ' FCC phase with an ordered structure are formed. The γ' phase is the primary strengthening phase in nickel based superalloys. The mechanical properties of these materials will strongly depend on the alloy microstructure, i.e. mainly on the chemical composition, the volume fraction and morphology of γ' particles.

Nickel based superalloys rich in cobalt owe their hardness and creep resistance to the presence of a high volume fraction (around 60%) of γ' particles with composition

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$(Ni,Co)_3(Al,Ti)$ which are coherent with the solid solution γ matrix containing Ni and Co, as well as Cr, Mo, W and low amount of Al and Ti. In addition to nickel, considered as a dominant element, Ti and Al are essential chemical elements for formation of γ' precipitates. It should be noted that the composition range of Al and Ti is limited by the possible occurrence of the η phase, which weakens the alloy and causes a significant lowering of its mechanical characteristics [1].

Cobalt plays a crucial role in high temperature creep by reducing the solubility of Al and Ti in the matrix, thus increasing the γ' phase volume fraction [2] and hardness [3]. Furthermore, the solubility of carbon in the γ matrix is increased [4].

Chromium is necessary for the corrosion resistance and plays a role in the interaction between the precipitates and the matrix [5]. However, an excess of this element may provoke a deterioration of mechanical properties. The addition of heavy metals such as Mo and W improves the mechanical strength of superalloys by solid solution effect. The observed hardening is mainly due to the difference of atomic size in the solution.

Carbon contributes to the hardening of the superalloy matrix by reducing the stacking fault energy [6]. Carbon is also regarded as a complex agent due to the fact that it combines with most metallic elements forming various types of carbides despite of the fact that in most superalloys its percentage does not exceed 0.2 wt. %. Table 1 illustrates types of carbides in different nickel based superalloys as a function of total content of Mo and W.

Table 1. Carbides present in different superalloys

Superalloys	Mo+W wt. %	Primary carbides	Secondary carbides	Other carbides	Réf.
Inconel 600	1.7	MC	$M_{23}C_6$		[7]
Nimonic 115	2.0	MC	$M_{23}C_6$		[12]
Udimet 500	2.3	MC	$M_{23}C_6$		[3]
Udimet 700	2.4	MC	$M_{23}C_6$		[3]
Alloy 713C	2.6	MC	$M_{23}C_6$		[7]
Similar to Udimet 500	5.18	MC (M=Ti,Mo)	$M_{23}C_6$ (M=Cr,Mo)	M_6C (M=Mo)	This work

Precipitates such as primary MC carbides (TiC, NbC, MoC) and secondary carbides $M_{23}C_6$ ($Cr_{23}C_6$) are distributed in grains and at grain boundaries [7, 8]. During heat treatment the primary carbides may be broken down according to the following chemical reaction:

$$MC + \gamma \rightarrow M_{23}C_6 + \gamma' \quad (1)$$

The aim of this paper was to investigate the effect of heat treatment on the microstructure of the cobalt-rich nickel based superalloy similar to Udimet 500 by its chemical composition.

Experimental

Methods and Materials

Chemical composition

The studied superalloy which is used for the manufacture of gas turbines blades is rich in cobalt (29 wt. %). The applied heat treatment was similar to the one used for industrial superalloys with chemical composition similar to that of Udimet 500. Several samples were collected after each stage of heat treatment in order to investigate its effect on the microstructure of superalloy. The mean chemical composition of this superalloy is presented in Table 2 [4].

Table 2. Chemical composition of studied superalloy (in wt. %)

Ni	Co	Cr	Al	Mo+W	Ti	Fe	Si	Mn	C
40.44	29.03	16.28	6.65	5.18	2.60	0.26	0.78	0.07	-

Microstructural characterization

The microstructure was studied by scanning electron microscope (SEM) and transmission electron microscope (TEM). In order to observe the microstructure by SEM, samples were ground with abrasive paper of different sizes from 180 to 1200, and then polished with the lubricant and diamond paste with the finesse up to 1 µ. After each polishing, the samples were rinsed with distilled water and dried. As a final stage of metallographic preparation, the samples, the discs with 3 mm diameter**,** were cut from the foil with an abrasive slurry cutting saw, mechanically ground to 150 µm thickness, and then electropolished with a solution of 10% acetic acid and perchloric acid at 10°C in a Struers Tenupol – a double-jet polisher. Thin foils were investigated by Philips EM 300 TEM microscope operating under an accelerating 200 kV voltage and equipped with a goniometric plate. A SEM JEOL J.S.M. - 225 was also used for microstructural investigations.

Table 3. The effect of heat treatment on appearance of microstructural features

Microstructures		Heat Treatment		
		1	1+2	1+2+3
In the matrix	Small γ' precipitates	* ↘		
	Coalesced γ' precipitates		↘ →	→
	Primary carbides MC (M=Ti, Mo)	→ ↘	→	→
At grain boundaries	Secondary carbides $M_{23}C_6$ (M=Cr, Mo)		↘ →	→

1- Solution treated 2h at 1150°C in argon atmosphere and oil quenched.
2- Aged for 24h at 843°C in vacuum.
3- Aged for 20h at 703°C in vacuum.

*In three columns of the right side of Table 3 the presence of the microstructural features obtained at the end of each sequence of the heat treatment is shown by a point with the small arrow. The longer arrows indicate the transition of these features from one stage of the heat treatment to the following stage.

Heat treatment

Heat treatment was conducted in a vertical furnace under controlled atmosphere of argon. Annealing for 2h at 1150°C was followed by quenching in order to retain the high temperature microstructure. The silicone oil was chosen as a quenchant with the aim to prevent the formation of additional carbides which may be formed if the mineral oil was used.

Three successive heat treatments are summarized in Table 3, with their effect on the appearance of microstructural features and their morphology.

Results and discussion

Influence of heat treatment

The heat treatment of superalloys applies in order to obtain adequate microstructure which will ensure optimal mechanical properties at high temperatures. As a first step the heat treatment is usually performed in a single phase range at temperatures between 1040-1230°C. The role of the high temperature homogenization heat treatment is to obtain a uniform γ matrix which will serve as a starting microstructure for the following intermediate heat treatments at lower temperatures. A number of intermediate heat treatments could be undertaken in order to obtain the optimal microstructure to ensure the best creep properties [9].

The most usual heat treatment for Udimet 500 (alloy close to superalloy studied in this work) consists of the high temperature homogenizing annealing at 1150°C for 2h and quenching followed by two intermediate heat treatments [10]. According to Terzi [9] this type of treatment provides optimal properties of hardness and tensile strength, but it leads to susceptibility to intergranular corrosion [11].

In this paper three separate heat treatments were applied (see Table 3), with their effect on the microstructure. Because the oil quenching was not sufficiently rapid, a relatively high volume fraction of γ‘particles was already present after the solution treatment.

TEM micrograph (Figure 1) shows the homogeneous distribution of the γ' phase particles in the γ matrix after quenching.

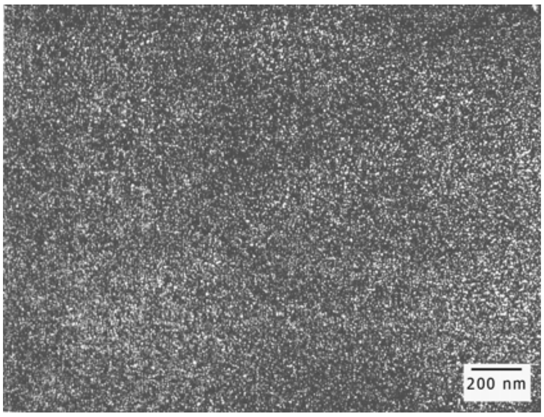
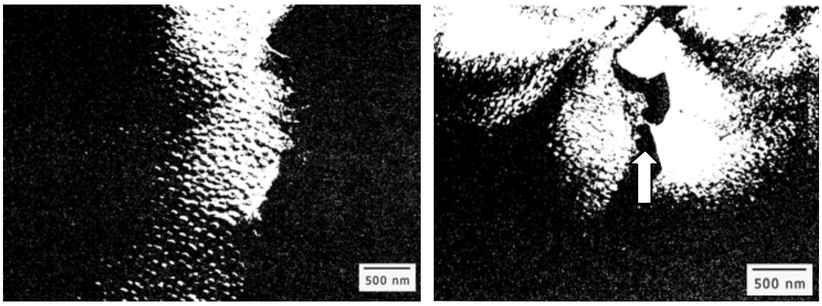


Figure1. TEM micrograph. The γ′ precipitates in the γ matrix after quenching from 1150°C/2h.

The aim of the first intermediate heat treatment (aging), i.e. the second stage (at 843°C for 24h) was to increase the size of γ' particles and to enable precipitation of carbides at the grain boundaries. Figure 2 (a, b) shows bright and dark TEM images with γ' precipitates having a spheroid shape as well as the carbides at the grain boundaries.



a *b*

Figure 2. TEM micrograph. Bright image of a sample quenched from 1150°C/2h followed by the second stage of heat treatment 843°C/24h. The secondary carbides (Table 3) at the grain boundaries are shown with the arrow.

Small γ' particles formed during the second stage of treatment do not coalesce at this relatively low temperature. The γ'particles are of the spherical shape with an approximate mean diameter of 65 nm. Their volume fraction was not measured. After the first ageing secondary carbides are precipitated at grain boundaries according to equation (1) at the expense of the primary carbides and the matrix [12].

The third stage of heat treatment at 703°C for 20h was aimed to obtain finer precipitation of γ' particles. Figure 3 shows the precipitation of γ' phase during this treatment.

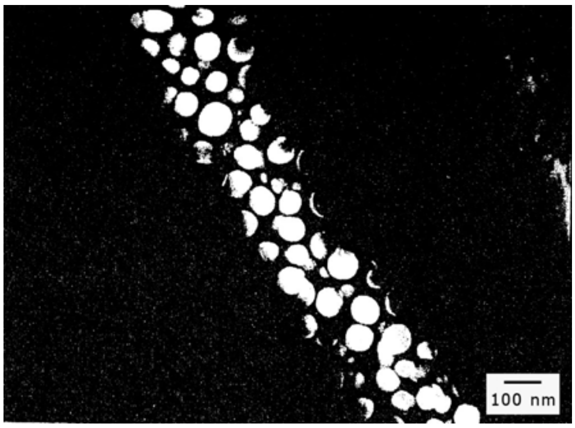
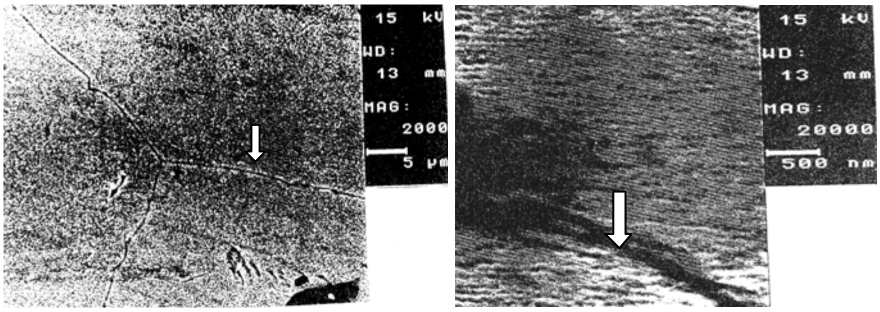


Figure 3. TEM micrograph. The microstructure of the sample after the third (final) stage of heat treatment: quenching from 1150°C/2h + 703°C/20h.The γ' phase precipitation.

SEM micrograph shows the precipitation of carbides at the grain boundaries after the final treatment (Figure 4 a, b).



a *b*

Figure 4: SEM micrograph. Microstructure after final processing. Note the heavy precipitation of the secondary carbides (see Table 3) at the grain boundaries (shown with the arrow).

Primary carbides are present during three stages of heat treatments. Secondary carbides were formed during two intermediate heat treatments.

Conclusion

The effect of heat treatment on the formation and precipitation of different microstructural features in a superalloy similar to Udimet 500 was studied by SEM and TEM. The same heat treatment as recommended for Udimet 500 was applied in this study. This type of treatment is composed of the high temperature homogenizing annealing at 1150°C for 2h and quenching followed by two separate intermediate heat treatments: (a) aging at 843°C for 24h, (b) aging at 703°C for 20h.

The cooling rate during quenching was not efficient to prevent the precipitation of a relatively high amount of γ' particles in the γ matrix. The observation of the structure performed after each step of the heat treatment revealed the presence of primary carbides during this treatment. Furthermore, a large precipitation of secondary carbides was observed at the grain boundaries as a result of intermediate heat treatments. The microstructure observed by TEM showed the precipitated γ' strengthening particles.

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