INFLUENCE OF COOLING RATE ON γ′ MORPHOLOGY IN CAST Ni – BASE SUPERALLOY

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Abstract
The Ni – base superalloys, which combine unique physical and mechanical properties, are used in aircraft industry for production of aero engine most stressed parts, as are turbine blades. From this reason a dendrite arm spacing, carbides size and distribution, morphology, number and value of γ′ - phase are very important structural characteristics for blade lifetime prediction as well as aero engine its self. In this article methods of quantitative metallography (software LUCIA for carbides evaluation, measuring of secondary dendrite arm spacing and coherent testing grid for γ′ - phase evaluation) are used for evaluation of structural characteristics mentioned above on experimental material – Ni base superalloy ŽS6K. The high temperature effect represented here by heat treatment at 800°C followed with holding time about 10 hours, and cooling rate, here represented by three various cooling mediums as water, air, and oil, on structural characteristics and application of quantitative methods evaluation are presented in this paper.

Keywords: nickel alloy, carbides and γ′ - phase metallographic evaluation, annealing, cooling rate influence.

1 Introduction

Aerospace industry is one of the biggest consumers of advanced materials, because of its unique combination of mechanical, physical properties and chemical stability. High alloyed stainless steel, titanium alloys and nickel base superalloys are most used for aerospace applications. High alloyed stainless steel is used for shafts of aero engine turbine, titanium alloys for compressor blades and finally nickel base superalloys are used for most stressed parts of jet engine – turbine blades. Nickel base superalloys were used in various structure modifications: as cast polycrystalline, directionally solidified, single crystaled and in last years materials produced by powder metallurgy [1]. In this paper problems of polycrystalline nickel base superalloys turbine blades such as most stressed parts of aero jet engine will be discussed.

The structure of polycrystalline Ni – base superalloys, depending on a heat – treatment, consist of solid solution of elements in Ni (γ - phase, also called matrix), primary carbides MC type (created by element such as Cr and Ti), intermetallic precipitate $Ni_3(Al, Ti)$ (γ′ - phase), and secondary carbides $M_{23}C_6$ type (created by elements such as Cr, Co, Mo, W). Shape and size of these structural components have a significant influence on final mechanical properties of alloy.

For instance the precipitate γ′ size greater than 0.8 μm significantly decreasing the creep rupture life of superalloys and also carbides size greater than 5 μm is not desirable because of fatigue cracks initiation [2].
For this reason needs of new non – conventional structure parameters methods evaluation were developed. The quantitative metallography, deep etching, and colour contrast belongs to the basic methods. The quantitative metallography analysis has statistical nature. The elementary tasks of quantitative metallography are:

- Dendrite arm spacing evaluation;
- Carbide size and distribution;
- Volume ratio of evaluated phase;
- Number ratio of evaluated phase;
- Size of evaluated phase.

Application of the quantitative metallography and colour contrast on the Ni – base superalloys are the main objectives discussed in this paper. More detailed analysis is published in previous works [1-11]. These non – conventional methods were successfully used also for the other types of materials [12-15].

2 Material and experimental methods

The cast Ni – base superalloy ŽS6K was used as an experimental material. Alloy ŽS6K contains higher amount of Cr, it has increased gas corrosion resistance and also high creep rupture life.
This alloy was evaluated after annealing at 800 °C/ 10 and 800 °C/ 15 hrs. and followed by cooling with various rate, presented with cooling in water, oil and air. The chemical composition in wt % is presented in **Table 1**.

Table 1 Experimental alloy chemical composition

Alloy	Elements (wt. %)								
	C	Ni	Co	Fe	Ti	Cr	Al	W	Mo
ŽS6K	0.13 - 0.2	base	4.0 - 5.5	2.0	2.5 - 3.2	9.5 - 12	5.0 - 6.0	4.5 - 5.5	3.5 - 4.8

*other minor elements: Mn, Si – 0.4; P, S, Ce, Bi – 0.015; B – 0.02; Pb – 0.005

Alloy ŽS6K is after casting strengthened with solid solution (Cr, Co and Mo), γ′ phase ($Ni_3(AlTi)$) and with carbides $M_{23}C_6$ situated on grain boundaries.
For evaluation of structural characteristics the following quantitative metallography methods were used:

- Carbide distribution and average size was evaluated by software LUCIA Metalo 5.0;
- Secondary dendrite arm spacing measurement;
- For number of γ′ - phase particles coherent testing grid with area probe of square shape were used;
- For volume of γ′ - phase particles coherent testing grid with 50 dot probes made of backslash crossing were used.

3 Results and discussion

The ŽS6K microstructure of starting stage is created by carbides in chain morphology situated on grain boundary and large amount of eutectic cells γ/γ′ (**Fig. 1**). An example of microstructure after annealing at 800 °C/ 15 hrs., focused on carbide distribution is presented in **Fig. 2**.

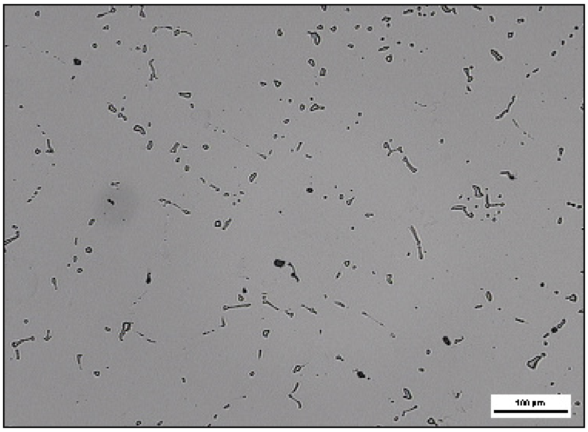


Fig.1 SuperalloyŽS6K: starting stage.

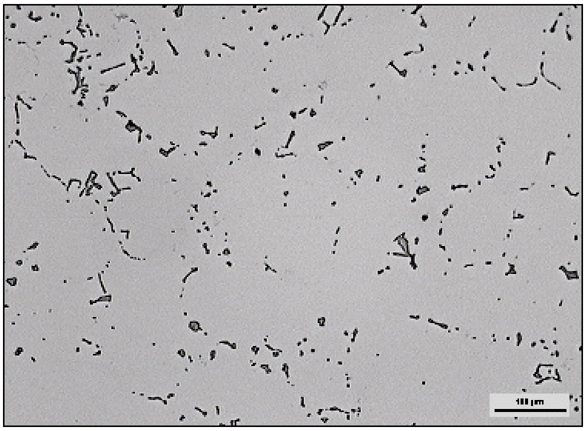


Fig.2 Superalloy ŽS6K: 800°C/15hrs. cooled in oil.

After 800 °C/ 10 and 800 °C/ 15 hrs. the microstructure shows some changes, mainly in number of carbides, its distribution and size. This effect is forced by diffusion mechanism and cooling rate when quick cooling represented by water gives not sufficient time for carbide growth. The results of carbide evaluation are presented in **Fig. 3**.

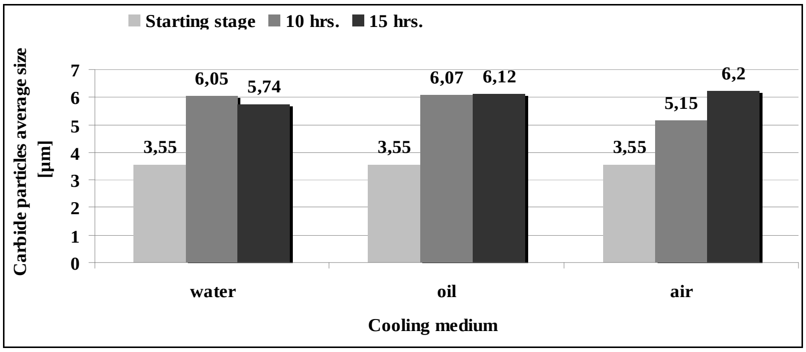


Fig.3 Carbide particles ratio depended from cooling medium and time of holding.

For dendrite structure evaluation method of measuring secondary dendrite arm spacing was used. The results of measuring are presented in **Table 2**. The cast materials are characterized by dendritic structure, as can be seen in **Fig. 4 a** and **4 b**, which is a result of chemical heterogeneity. Increase of annealing time decreases its chemical heterogeneity. It means that the secondary dendrite arm spacing is increased (the dendrites are growing). ŽS6K dendrite arm spacing is increased in dependence of the annealing time, annealing temperature and cooling medium from 113.64 to 156.25 μm.

a) 

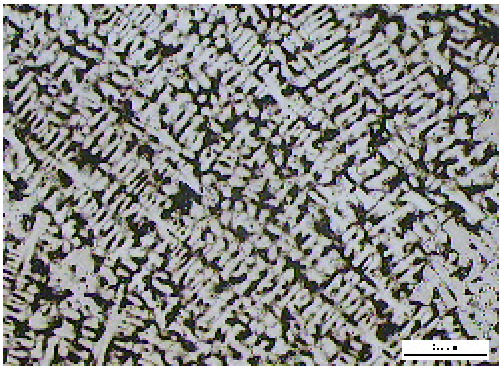
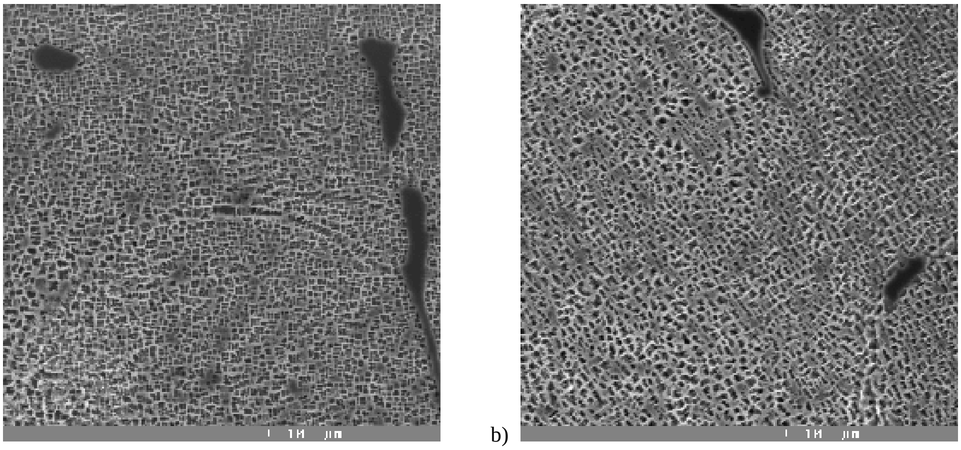
b) 

Fig.4 Superalloy ŽS6K, secondary dendrite arm spacing: a) starting stage, b) 800 °C/ 10 hrs. cooled in water, etch. MARBLE

Table 2 The results of secondary dendrite arm spacing evaluation

Secondary dendrite arm spacing [μm]					
Alloy	Air	Oil	Water	Starting stage	
ŽS6K 10h	138,89	131,58	126,58	ŽS6Kv	185,19
ŽS6K 15h	156,25	131,58	113,64		

The characteristics of γ´ - phase morphology were also measured using the coherent testing grid methods. As were mentioned above, the number and volume of γ´ - phase have significant influence on mechanical properties of this alloy, especially on creep rupture life. Average satisfactory size of γ´ - phase is about 0.35 – 0.45 μm and also carbide size should not exceed size of 5 μm – because of fatigue crack initiation [3]. Another risk of using high temperature loading or annealing is creation of TCP phases, such σ - phase or Laves phase, in range of temperature 750 °C – 800 °C. Exposing for 10 hours at annealing temperature the volume of γ' - phase was increased about 16.8 – 33 % comparing with the starting stage, **Fig. 5 a** and **5 b**. The significant increasing of γ' - phase was observed at holding time 15 hours, cooling on air, where volume of γ' - phase is 76.6 %.



a) b)

Fig.5 Superalloy ŽS6K: a) 800°C/10 hrs. cooled in water, b) 800 °C/ 15 hrs. cooled in water, etch. MARBLE

Vickers hardness measuring was carried out to confirm possible carbide re-distribution and decreasing of the chemical heterogeneity. It was found that the main influence on the hardness variation has cooling medium, as we can see from results presented in **Table 3**. The highest hardness was reached after cooling on air, after 15 hours – 493 HV 10. Cooling in oil, after 10 hours, brings to hardness 476 HV 10 and the lowest hardness was measured for the water cooling – 443 HV 10. From the result it is clear that holding time at temperature of annealing has no so significant effect on hardness value, but the cooling rate represented by various cooling medium has significant influence on the hardness.

Table 3 The γ' - phase morphology evaluation including Vickers hardness measuring

Alloy	Number of γ' - phase N [μm^{-2}]	Volume of γ' - phase V [%]	Size of γ' - phase u [μm]	Average carbide size [μm]	Hardness [HV 10]
ŽS6Kv	2,47	39,4	0,61	3.55	408,6
ZŠ6K10h water	1,95	56,2	0,54	6.05	456,70
ŽS6K10h oil	1,60	63	0,63	6.07	476,31
ŽS6K10h air	1,50	72,4	0,69	5.15	489,67
ŽS6K15h water	1,90	66,8	0,59	5.74	443,86
ŽS6K15h oil	1,59	71,8	0,67	6.12	463,05
ŽS6K15h air	1,49	76,6	0,72	6.2	493,73

4 Conclusion

As cast Ni – base superalloy ŽS6K was used as an experimental material. The structural characteristics were evaluated from starting stage of sample and after annealing at 800 °C/ 10 and 800 °C/ 15 hrs. with using of quantitative metallography methods. The results are as follows:

- Structure of the samples is characterized by dendritic segregation. In dendritic areas fine γ' - phase is segregate. In interdendritic areas eutectic cells γ/γ' and carbides are segregated.
- Holding time (10 – 15 hrs.) does have significant influence on the carbide particles size. The size of carbides is under critical level for fatigue crack initiation only in starting stage. The increase rate of cooling has significant effect on the carbide particles ratio.
- Chemical heterogeneity of the samples with longer holding time is decreasing. It is reason of sufficient time for diffusion mechanism, which is confirmed by secondary dendrite arm spacing measurement results.
- The volume of γ' - phase with longer holding time is increasing and also γ' - phase size is growing. With higher rate of cooling are γ' particles finer.
- There was not evidence of TCP phase presence even though high annealing temperature.
- Cooling rate has also influence on the hardness. At lower rate of cooling the internal stresses are relaxed, which caused hardness increase – changing of the dislocation structure.

Cooling rates, represented by various cooling mediums have influence on diffusion processes in structure of alloy. These diffusion processes are the main mechanism for segregation and creating of carbide particles, equalization of chemical heterogeneity, γ' - phase segregation and are responsible for structure degradation of this alloy as well.

Acknowledgements

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