QUANTITATIVE METALOGRAPHY OF HEAT TREATED ŽS6K SUPERALLOY

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Resume
Alloy ŽS6K is former USSR superalloy used in DV – 2 jet engine. It is used for turbine rotor blade and whole cast small sized rotors with working temperature up to 800 ÷ 1050°C. This alloy was evaluated after annealing at 800 °C/ 10 and followed by cooling with various rate, presented with cooling in water, oil and air. Cooling rates, represented by various cooling mediums, have a significant influence on diffusion processes, which are going in structure. Methods of quantitative metallography (Image Analyzer software NIS – Elements for carbides evaluation, measuring of secondary dendrite arm spacing and coherent testing grid for γ′ - phase evaluation) are used for evaluation of structural characteristics on experimental material – Ni base superalloy ŽS6K.

Available online: http://fstroj.uniza.sk/PDF/2011/21-2011.pdf

Article info
Article history:
Received 22 July 2011
Accepted 17 September 2011
Online 28 September 2011

Keywords:
ŽS6K superalloy
Annealing
Degradation
Diffusion
Quantitative analysis

ISSN 1335-0803

1. Introduction

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 crystal and in last year’s 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 [2]. 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 50 μm is not desirable because of fatigue cracks initiation [3].

For this reason needs of new non – conventional structure parameters methods evaluation were developed. 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 gamma prime phase;
- Number ratio of evaluated gamma prime phase;
- Size of evaluated gamma prime 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 [1-9]. These non – conventional methods were successfully used also for the other types of materials [10-15].

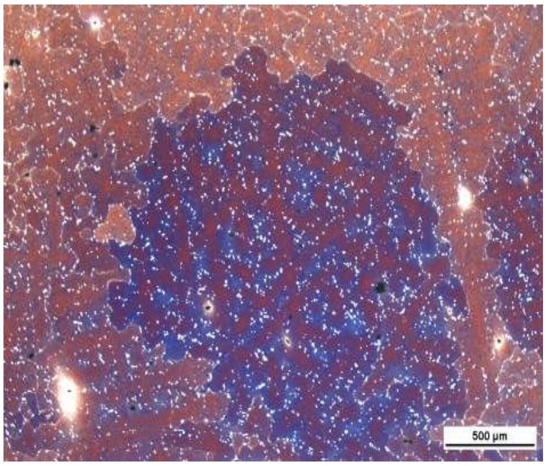
2. Experimental

2.1. Experimental material

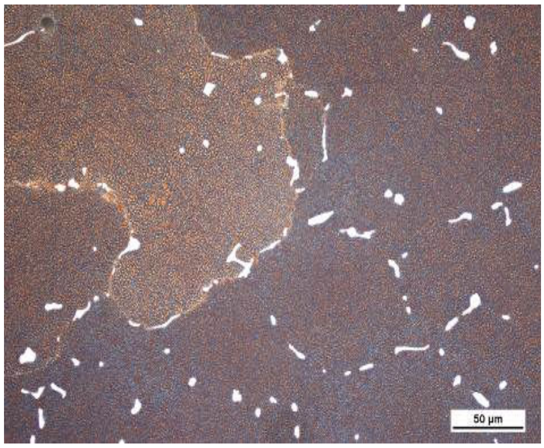
The cast Ni – base superalloy ŽS6K was used as an experimental material. Alloy ŽS6K is former USSR superalloy used in DV – 2 jet engine. It is used for turbine rotor blade and whole cast small sized rotors with working temperature up to 800 ÷ 1050°C. The alloy is made in vacuum furnaces. Parts are made by method of precise casting. Temperature of liquid at casting in vacuum to form is 1500 ÷ 1600°C, depends on parts shape and its amount. Cast ability of this alloy is very well with only 2 ÷ 2.5% of shrinkage. Blades made of this alloy are also protected against hot corrosion with protective heat proof alitize layer, so there are able to work at temperatures up to 750°C for 500 flying hours.

This alloy was evaluated after annealing at 800 °C/ 10 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.

A typical microstructure of ŽS6K Ni – base superalloy as – cast is showed in Fig. 1. and 2. Microstructure of as – cast superalloy consist of significant dendritic segregation caused by chemical heterogeneity (Fig. 1a) and particles of primary MC and secondary $M_{23}C_6$ carbides (Fig. 1b). Primary carbides (Ti, Mo, and W)C are presented as an block shape particles mainly inside of grains. Secondary carbides are presented as a "Chinese" script shape particles on grain boundaries.



a) dendritic segregation



b) MC and $M_{23}C_6$ carbides

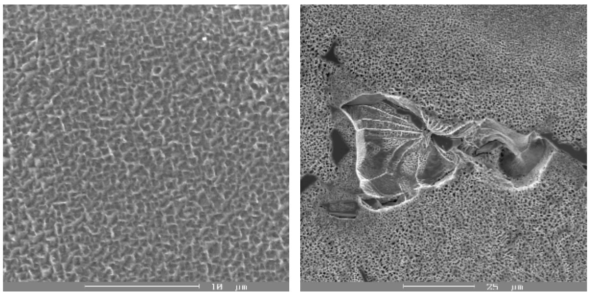
Fig. 1. Microstructure of as – cast Ni – base superalloy ŽS6K, Beraha III
(full colour version available online)

Table 1

Chemical composition of ŽS6K alloy (in wt. %)

C	Ni	Co	Ti	Cr	Al	W	Mo	Fe	Mn	P	S	Pb	Bi
0.13 ÷ 0.2	Bal	4.0 ÷ 5.5	2.5 ÷ 3.2	9.5 ÷ 12	5.0 ÷ 6.0	4.5 ÷ 5.5	3.5 ÷ 4.8	2	0.4	0.015	0.015	0.001	0.0005

However, microstructure also contains solid solution of elements in base nickel matrix – so called γ phase (Ni(Cr, Co, and Fe)) and strengthening phase, which is product of artificial age – hardening and has significant influence on mechanical properties and creep rupture life – so called γ′ phase (gamma prime, $Ni_3(Al, and Ti)$), Fig. 2a. Of course, both of these phases, γ (gamma) and γ′ (gamma prime) are creating an eutectic γ/γ′, Fig. 2b.



a) matrix and γ′ phase *b) γ/γ′ eutectic*

Fig. 2. Microstructure of as – cast Ni – base superalloy ŽS6K, Marble, SEM

2.2 Experimental methods

For evaluation of structural characteristics the following quantitative metallography methods were used:

- Carbide distribution and average size was evaluated by software NIS - Elements;
- Secondary dendrite arm spacing measurement;
- For number of γ′ - phase particles coherent testing grid with 9 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.

Secondary dendrite arm spacing was evaluated according to Fig. 3. and calculated with formula (1). Changing of distance between secondary dendrite arms "d" is important characteristic because of base material; matrix γ, degradation via equalizing of chemical heterogeneity and also grain size growing.

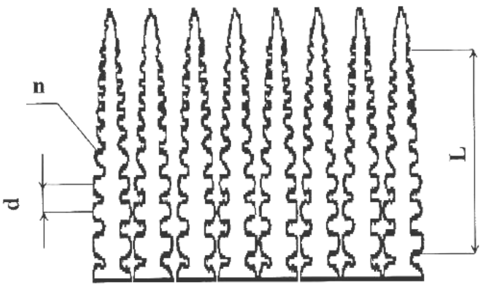
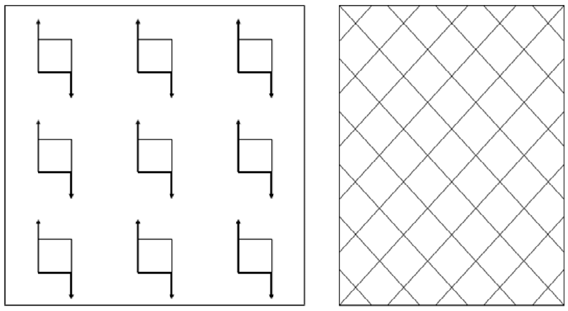


Fig. 3. Scheme for secondary dendrite arm spacing evaluation

$$d = \frac{L}{n} \cdot \frac{1}{z} \cdot 1000 \quad (\mu m) \qquad (1)$$

For evaluation of γ and γ′ phases were used method of coherent testing grid, number of γ′ "N" has been evaluated by grid with 9 area probes of square shape (Fig. 4a) and volume of γ′ "V" has been evaluated by grid with 50 dot probes (Fig. 4b). After measurement was values calculated with formulas (2) and (3). Size of γ′ is also important from creep rupture life point of view. Precipitate with size higher than 0.8 μm can be considered as heavy degraded and cause decreasing mechanical strength at higher temperatures.



a) number of γ′ particles *b) volume of γ′ particles*

Fig. 4. Coherent testing grid for γ′ evaluation

$$N = 1.11 \cdot z^2 \cdot x_{str} \cdot 10^{-9} \quad (\mu m^{-2}) \qquad (2)$$

$$V = 2 \cdot n_s \quad (\%) \qquad (3)$$

3. Experimental results and discussion

As a first characteristic were carbide size and its distribution evaluated. There were compared specimens made of ŽS6K superalloy at starting stage and after 800°C/10 hrs.

Cooling rate depends from cooling medium; in our case were air, oil, and water used. Results for ratio of carbide particles in observed area are in Fig. 5. and results from average carbide size are in Fig. 6.

From presented relations (Fig. 6) is obvious that holding time on various temperatures of annealing and cooling in selected mediums does not have significant influence on carbide particle size. More significant influence on ratio of carbide particles has cooling rate (Fig. 5). With increasing speed of cooling and longer holding time on annealing temperature is carbide particles ratio decreasing.

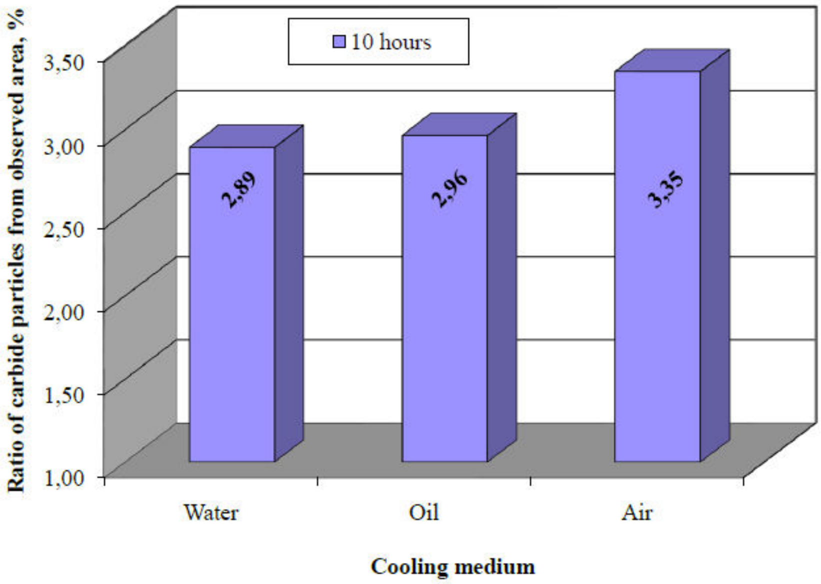


Fig. 5. Ratio of carbide particles from observed area

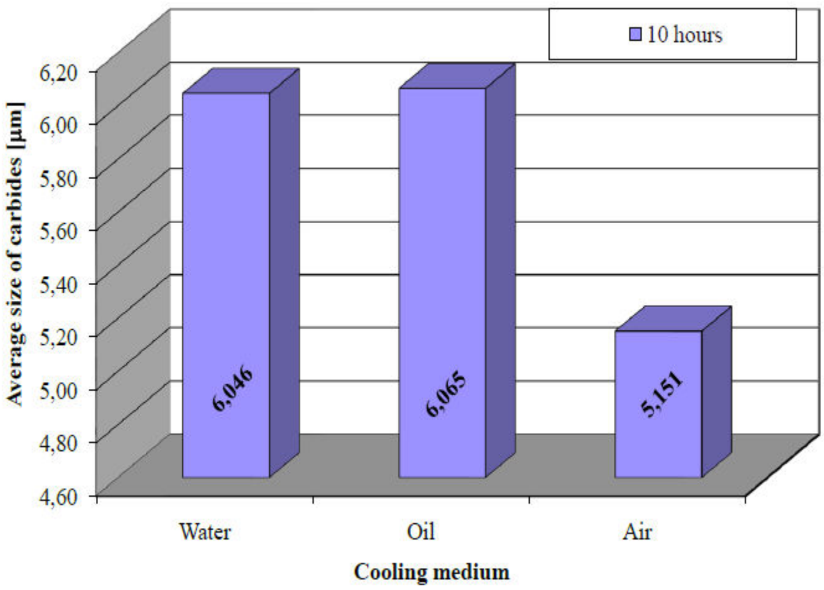
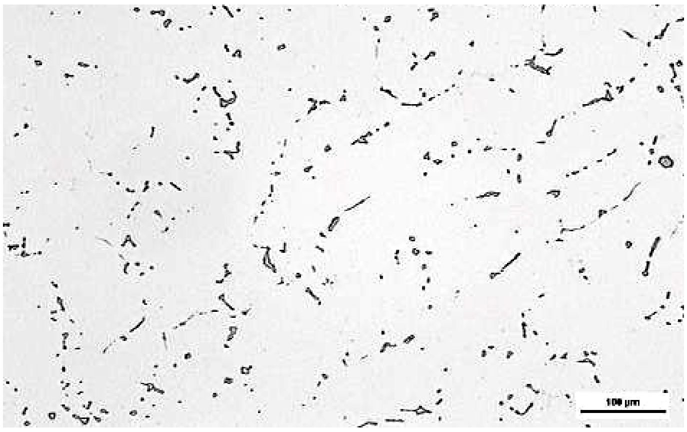


Fig. 6. Average carbides size (μm)

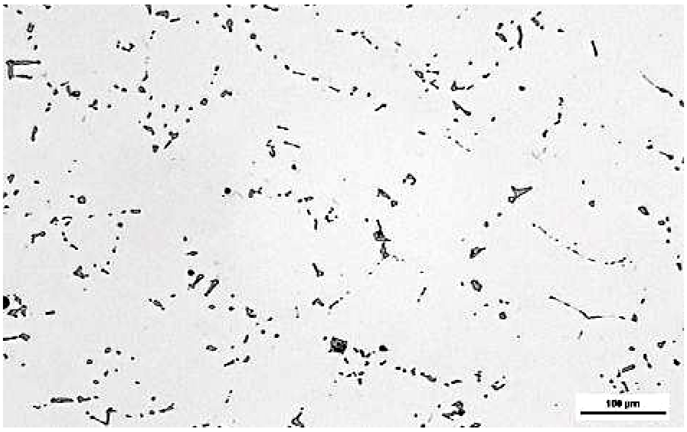
Generally, we can suppose, that with temperature of annealing are carbide particles partially dissolved and elements, which are consider as an carbide creators (in this case mainly Ti) have create a new particles of γ' phase. This phenomenon has influence on decreasing of segregated carbide percentage ratio. With increasing of cooling rate (water, oil) an amount of γ' phase has decreased and carbides percentage ratio is higher. At slow cooling and longer time of holding is segregate higher amount of γ' and therefore ratio of carbides decrease. It is all happen according to scheme:

$$MC + \gamma \rightarrow M_{23}C_6 + \gamma'$$

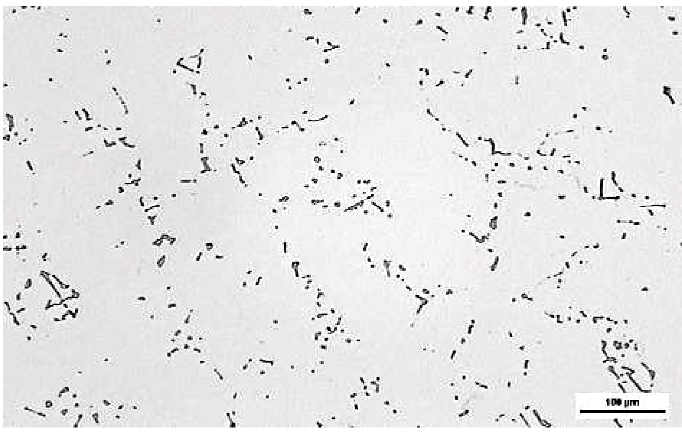
Microstructures equivalent to this evaluations are on Fig. 7. For carbide evaluation is etching not necessary. All micrographs are none etched.



a) water cooling

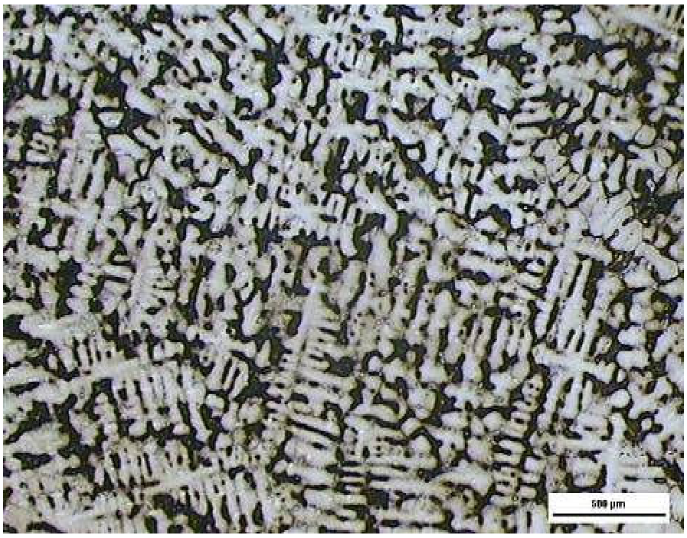


b) oil cooling,

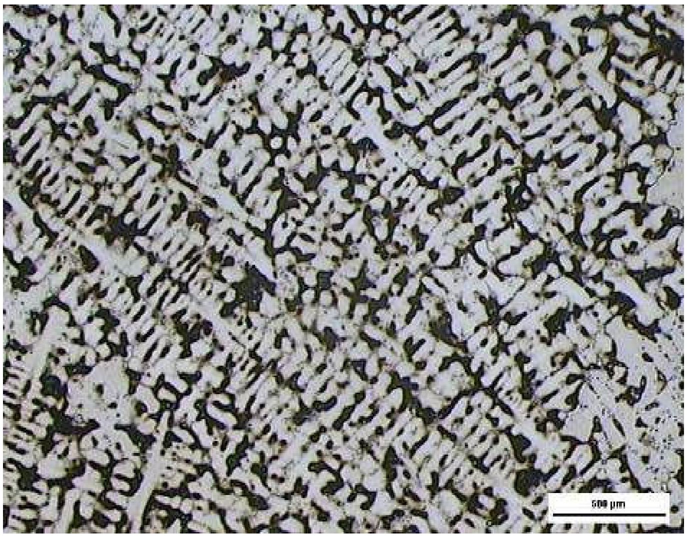


c) air cooling

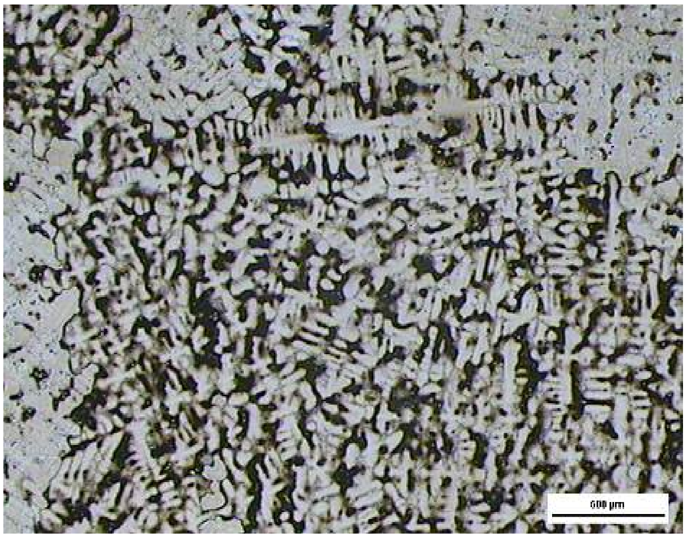
Fig. 7. Microstructure of ŽS6K, carbides ratio after 800°C annealing/10 hrs.



a) water cooling



b) oil cooling



c) air cooling, etch. Marble

Fig. 8 Dendritic segregation of ŽS6K, 800°C/10 hrs.

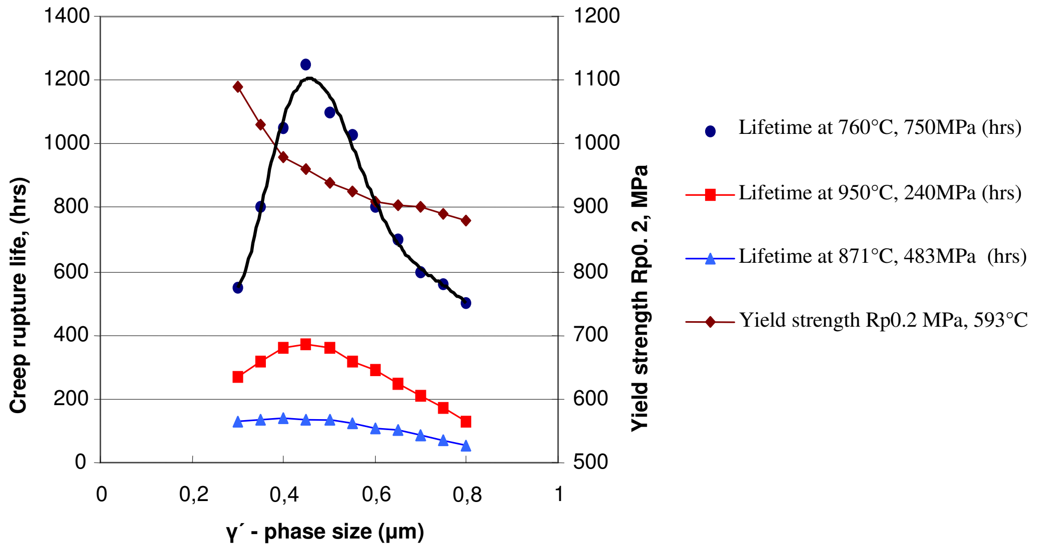


Fig. 9. Gamma prime size and its influence on chosen mechanical characteristics at various temperatures

The second characteristic what has been evaluated is dendrite arm spacing, as it is shown in Fig. 3. Results are in Table 2.

Table 2

Results from secondary dendrite arm spacing (μm)

ŽS6K – starting stage	185.19		
Cooling medium	**Water**	**Oil**	**Air**
ŽS6K/10hrs.	126.58	131.58	138.89

Cast materials are characteristic with dendritic segregation, which is caused by chemical heterogeneity. With influence of holding at annealing temperature is chemical heterogeneity decreasing. It means, that distance between secondary dendrite arms is increasing (dendrites are growing). From results mentioned above (Table 2) is clear to see that with higher cooling rate comes to slowing of diffusion processes and dendrite arm spacing is decreasing in comparing with starting stage, Fig. 1a. All these changes are also obvious on Fig. 8.

ŽS6K dendrite arm spacing is increased in dependence of the annealing time, annealing temperature and cooling medium from 126.58 to 138.89 μm.

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 (Fig. 9) 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.

Results from γ´ - phase evaluation with using of coherent testing grid are listed below in Table 3.

Table 3

Results from γ´ - phase evaluation

Cooling medium	**Number of γ´ - phase N (μm^{-2})**	**Volume of γ´ - phase V (%)**	**Average size of γ´ - phase u (μm)**
Start. stage	2.47	39.4	0.61
10h water	1.95	56.2	0.54
10h oil	1.60	63	0.63
10h air	1.50	72.4	0.69

4. Conclusions

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/ 10hrs. 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 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 no 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 a significant influence on diffusion processes, which are going in structure. These diffusion processes are main mechanism for segregation and carbide particles forming, equalize of chemical heterogeneity (represented by dendrite arm spacing), segregation of γ' - phase and as well as are responsible for structure degradation of such alloy.

Acknowledgements

This work has been supported by Scientific Grant Agency of Ministry of Education of Slovak Republic N°1/0249/09, N°1/0841/11, N°1/0193/10, 220-009ŽU-4/2010 and SK-CZ-0086-09.

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