ISSN 1392–1320 MATERIALS SCIENCE (MEDŽIAGOTYRA). Vol. 14, No. 4. 2008

Structural Analyses of Advanced Material for Aerospace Industry

Juraj BELAN[*]

Department of Materials Engineering, University of Žilina, Univerzitná 1, 01026 Žilina, Slovakia

Received 29 May 2008; accepted 11 September 2008

The Ni – base superalloys 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 are used methods of quantitative metallography (software LUCIA for carbides evaluation, measuring of secondary dendrite arm spacing and coherent testing grid for γ' - phase evaluation) for evaluation of structural characteristics mentioned above on experimental material Inconel IN 738. The high temperature effect and cooling rate on structural characteristics and application of quantitative methods evaluation are presented in this paper.

Keywords: Ni – base superalloys, turbine blades, quantitative metallography, γ' - phases.

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, consists 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, 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 – 7]. These non – conventional methods were successfully used also for the other types of materials [8 – 10].

2. EXPERIMENTAL

The cast Ni – base superalloy Inconel IN 738 was used as an experimental material. Alloy IN 738 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 hrs. 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	Nb	Ti	Cr	Al	W	Mo
IN 738	0.17	base	8.5	0.9	3.4	16.0	3.4	2.6	1.7

Alloy Inconel IN 738 is produced in two modifications as a low carbon (IN 738LC) and high carbon (IN 738C). After casting it is 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.

---

[*] Corresponding author. Tel.: +421-415-132611; fax.: +421-415-652940.
E-mail address: *juraj.belan@fstroj.uniza.sk* (J. Belan)

3. EXPERIMENTAL RESULTS AND DISCUSSIONS

The Inconel IN 738 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.

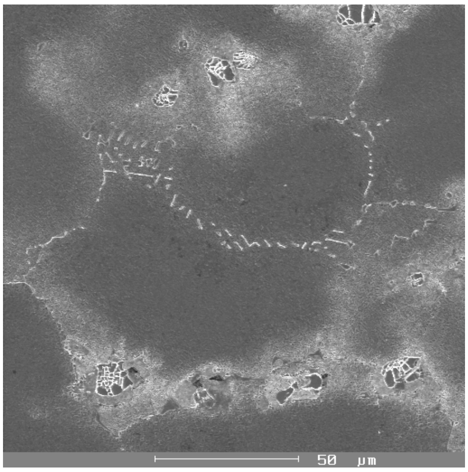


Fig. 1. Superalloy IN 738: starting stage, etch. Marble, SEM

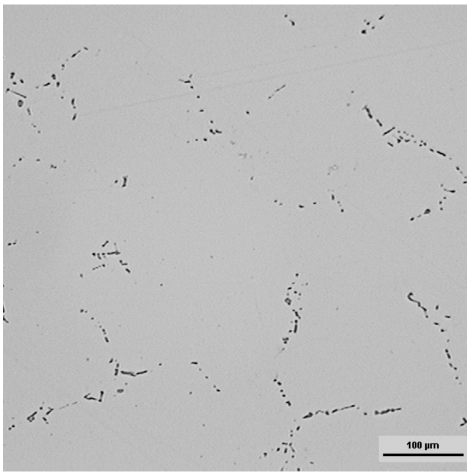


Fig. 2. Superalloy IN 738: 800 °C / 15 hrs. cooled in oil, etch. Marble

After 800 °C / 10 hrs. 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.

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). Inconel IN 738 dendrite arm spacing is increased in dependence of the annealing time, annealing temperature and cooling medium from 137 µm to 172 µm.

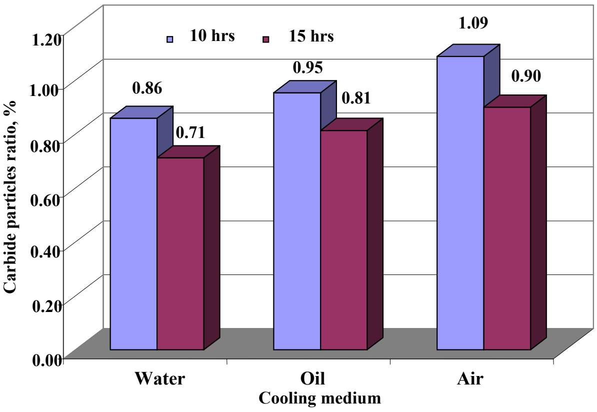
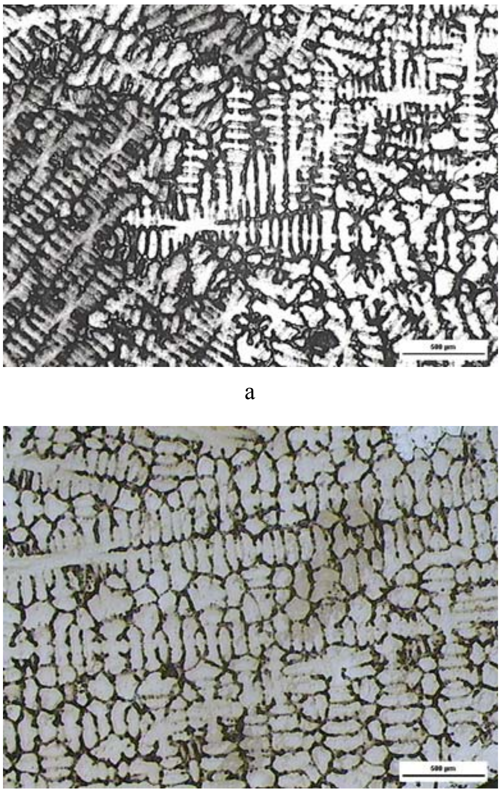


Fig. 3. Carbide particles ratio depended from cooling medium and time of holding



a

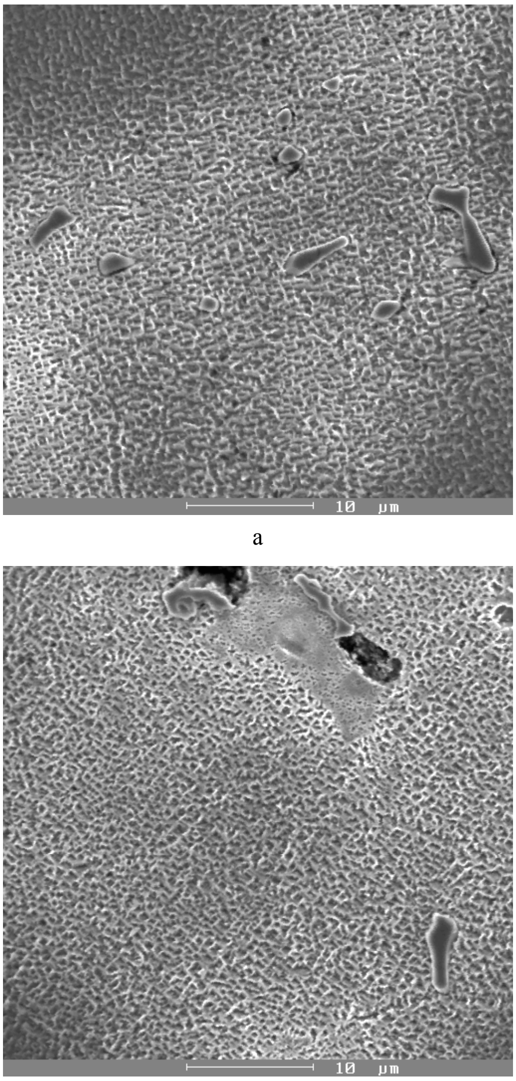
b

Fig. 4. Superalloy IN 738, 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	
IN738 10h	166.67	151.52	136.99	IN738v	161.29
IN738 15h	172.41	156.25	142.86		

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 µm – 0.45 µm and also carbide size should not exceed size of 5 μm – because of fatigue crack initiation [4]. 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 15 hours at annealing temperature the volume of γ′ - phase was increased about 2 % – 5 % comparing with the exposure time 10 hours, Fig. 5, a and 5, b. The significant increasing of γ′ - phase was observed at cooling on air where volume of γ′ - phase is 71.2 %.



a

b

Fig. 5. Superalloy IN 738: 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 in water – 413 HV 10. Cooling in oil brings to hardness 394 HV 10 and the lowest hardness was measured for the air cooling – 373 HV 10. From the result it is clear that holding time at temperature of annealing has no 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]
IN738v	1.68	60.4	0.76	–	–
IN738 10h water	2.14	58.4	0.52	3.629	374
IN738 10h oil	1.84	63.6	0.59	3.695	394
IN738 10h air	1.63	66.6	0.64	3.803	410
IN738 15h water	2.02	61.2	0.55	3.609	373
IN738 15h oil	1.84	65.8	0.60	3.774	394
IN738 15h air	1.58	71.2	0.67	3.608	413

3. CONCLUSIONS

As cast Ni – base superalloy Inconel IN 738 was used as an experimental material. The structural characteristics were evaluated from starting stage of sample and after annealing at 800 °C / 10 hrs. 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 hrs. – 15 hrs.) does not have significant influence on the carbide particles size. The size of carbides is under critical level for fatigue crack initiation. 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 higher rate of cooling the internal stresses are created, 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.

Acknowledgments

This work has been supported by Scientific Grant Agency of Ministry of Education of Slovak Republic and Slovak Academy of Sciences, grant No. 1/3153/06 “Structural fundamentals of high – loaded cast properties degradation“ and Culture and Educational Grant Agency of Ministry of Education of Slovak Republic, grant No. 3/6078/08 “Creation of a laboratory and textbooks for teaching of subject „Properties and using of materials“ and No. 3/4142/06 „Creation of laboratory and textbooks for teaching of subject „Materials quality control“. The part of the results of this work has been supported by SK – CZ international program No. 06K1201.

REFERENCES

1. **Gell, M., Duhl, D. N.** Progressive Technologies in Superalloys *Advanced High Temperature Alloys* ASM 1985: pp. 41 – 49.
2. **Copley, S. M., Kear, B. H.** Structural Characteristics of Superalloys. Part I *The Minerals, Metals & Materials Society – The American Institute of Mining, Metallurgical, and Petroleum Engineers* 239 1967: pp. 977 – 983.
3. **Copley, S. M., Kear, B. H.** Structural Characteristics of Superalloys. Part II *The Minerals, Metals & Materials Society – The American Institute of Mining, Metallurgical, and Petroleum Engineers* 239 1967: pp. 984 – 989.
4. **Donachie, M. J.** Superalloy Source Book. ASM, 1984: pp. 102 – 111.
5. **Leverant, G. R., Kear, B. H.** Mechanical Properties of Advanced Superalloys *Metallurgical and Materials Transactions* 1 1970: pp. 491 – 498.
6. **Jackson, J. J.** Evaluation of Superalloys Structural Characteristics *Metallurgical and Materials Transactions* 8A (10) 1977: pp. 1615 – 1620.
7. **Klacková, M.** Hidden History of Production *In: Productivity and Innovation* Slovak Productivity Center 03 2006: pp. 20 – 21 (in Slovak).
8. **Skočovský, P., Matejka, M.** Cast Iron Microstructure – Metallography Handbook. Fompex, Trenčín, ES VŠDS, 1994 (in Slovak).
9. **Skočovský, P., Vaško, A.** The Quantitative Evaluation of Cast Iron Structure. 1st Edition, EDIS, Žilina, 2007: 73 s. (in Slovak).
10. **Belan, J., Skočovský, P.** The Quantitative Metallography of Ni – Base Superalloys *XX Miedzynarodowe Sympozjum, Metody oceny struktury oraz wlasnosci materialow i wyrobow* Ustron – Jaszowiec, Poland, 2005: pp. 83 – 88.

Presented at the 17th International Conference
"Materials Engineering’2008"
(*Kaunas, Lithuania, November 06 – 07, 2008*)

DOI: 10.5755/j02.ms.26197