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Employing various metallography methods at high temperature alloy fatigue tests evaluation

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Abstract. Microstructures of superalloys have dramatically changed throughout the years, as modern technology of its casting or forging has become more sophisticated. The first superalloys have polyedric microstructure consisting of gamma solid solution, some fraction of gamma prime and of course grain boundaries. As demands on higher performance of aero jet engine increases, the changes in superalloys microstructure become more significant. A further step in microstructure evolution was directionally solidified alloys with columnar gamma prime particles. The latest microstructures are mostly monocrystalline, oriented in [001] direction of FCC gamma matrix. All microstructure changes bring necessity of proper preparation and evaluation of microstructure. Except for the already mentioned structures have gamma double prime and various carbides form can be seen. These structural parameters have mainly positive influence on important mechanical properties of superalloys. The paper deals with a microstructural evaluation of both groups of alloys – cast and as well as wrought. Microstructure evaluation helps to describe mechanism at various loading and failure of progressive superalloys. Such an example where microstructure evaluation is employed is fractography of failure surfaces after fatigue tests, which are examples of metallography evaluation described in this paper as a secondary objective.

Key words – Ni-base casting and wrought alloys, gamma prime, gamma double prime, gamma solid solution, light microscopy, scanning electron microscopy (SEM)

1. Introduction

The prime objective of metallography is to understand the relationship of composition, processing, and mechanical behaviour to the microstructure (RADAVICH J. F. 1997).

Superalloys are complex alloys of Fe-Ni, Ni-, and Co-base compositions. Their microstructure can be quite complex due to the potential for a variety of phases that can form in heat-treatment or service exposure conditions.

Preparation of superalloys for microstructural examination is not exceptionally difficult. The procedures are similar to those used to prepare stainless steels. Because they are face centred cubic (FCC) “austenitic” alloys with exceptionally good toughness, their machinability is poorer than for steel, and the age hardened alloys, especially the cast alloys, can be more difficult to section than most steel–when it has a very high γ'- phase content (VANDER VOORT G., MANILOVA E. P. 2004).

The whole metallography specimen preparation process consists of a few fundamental steps. The first step is cutting-off a specimen with proper dimensions (average surface size is 1 cm^2). Then the preparation is followed with a specimen mounting.

After mounting, preparation continues with grinding and polishing of specimens. Polishing usually commences with either 6 μm or 3 μm diamond abrasive, as a paste, slurry, or aerosol with the appropriate liquid extender/lubricant on a cloth pad. The first polishing step is followed with a second diamond abrasive step, generally 3 or 1 μm in size, or they used one or two steps with aqueous aluminium oxide slurries (www.georgevandervoort.com).

Numerous etchants are used to reveal the structure of superalloys. Some minor second-phase particles can be easily observed in the as-polished condition using bright field illumination. Obviously, non-metallic inclusions can be best observed in the as-polished condition.

However, even when such amount of etchant for superalloys is available, the use of the proper one depends on specific superalloy type (wrought or cast superalloy, differences in chemical composition, heat-treatment and so). It has been found that conventional mechanical polishing and immersion etching does not reveal equally well the various phases that can be present in alloy 718. In many cases depending on the thermal treatments, the corrosion behaviour of the alloy can necessitate stronger acid solutions or longer etching times or both (RADAVICH J. F. 1988). There are slightly different approaches for metallography of cast and wrought superalloys. For cast superalloys it is significant to reveal a dendritic microstructure to help evaluate the secondary dendrite arm spacing. On the other hand, wrought superalloys metallography evaluates the grain boundary and deformation, as well as crystallization twins.

The colour etching (SKOČOVSKÝ P., PODRÁBSKY T. 2001) is a quite novel method to reveal microstructure of superalloys and adds an interesting dimension into microstructure evaluation. Anyway, colour etching is not only way how to add some colour to microstructure. Using of DIC (Differential Interference Contrast) or Nomarski polarisers work well and gives satisfied results.

2. Experimental material and methods

The Ni-base superalloys in cast and wrought form were used as experimental materials. From cast superalloys group is alloy ŽS6K (which is former USSR cast superalloy) and the second group of wrought superalloys is Inconel's Alloy 718. The chemical composition of experimental materials is listed in Table 1.

Metallography specimens were prepared by cutting on MTH Micron 3000 precise saw, and mounting into bakelite mixture in Struers Cito-Press 1 and, finally, they were grinded and polished with of Struers Tegra-System (TegraPol-15 and TegraForce-1).

Table 1. The chemical composition of experimental superalloys (in wt. %)

Alloy	C	Co	Nb	Ti	Cr	Al	W	Mo	Fe	Mn
ŽS6K	0.2	5.5	-	3.2	12	6	5.5	4.8	2	0.4
Alloy 718	0.026	0.14	5.3	0.96	19.31	0.57	-	2.99	11.15	0.07

*Ni content is balance, Source: own study

Grinding and polishing consist of a few steps; grinding with SiC sand paper No. 320, followed by medium polishing with MD-Allegro and fine polishing with OP-S lubricant.

All the mentioned procedures for cast and wrought superalloys were used while preparing metallographic specimens after high frequency, and high cycle fatigue test via push-pull loading of IN 718 superalloy, as well as low frequency fatigue testing of three point bending load for the same superalloy.

Fatigue test at push-pull loading was done at frequency of f = 20 000 kHz at R = -1 and three point bending test was done at frequency f = 150 Hz with R = 0.11. For all fatigue tests number of cycles $N_f = 10^7$ were considered as fatigue lifetime limit σ_c. After tests also SEM fractography of fracture surfaces was performed.

3. Results of experiments

Microstructure of Ni-base superalloys is formed in dependence of chemical composition as follows:

Chromium, *Cobalt*, and *Ferrum* creates the substitution solid solution called γ- phase (also known as matrix). This phase has austenitic FCC lattice. Solid solution provides fundamental strength characteristics of superalloys (Belan J. 2012).

Aluminium and *Titanium* are considered as main elements responsible for hardening of solid solution via precipitation mechanism when forms γ'-phase ($Ni_3Al(Ti)$ gamma prime). Gamma prime phase is coherent to solid solution FCC matrix γ and has $L1_2$ lattice. Unique properties of superalloys are closely related to fraction of γ'-phase. Its optimum size for high-temperature application is 0.35 μm - 0.45 μm where superalloys combine satisfactory creep rupture strength and yield strength Rp0.2 (Donachie M. J., Donachie S. J. 2002).

Niobium and *Tantalum* when presented in superalloy at higher amount, may form hardening phase γ'' (Ni_3Nb gamma double prime), which is considered as main precipitation hardening phase in Alloy 718 type. Gamma double prime crystalizes in DO_{22} lattice and is characterized by disk-like shape with the small dimension aligned along the three [001] directions. In addition, there are some γ' precipitates present on which the particles γ'' have been shown to precipitate (Pineau A., Antolovich S. D. 2009).

Molybdenum and *Tungsten* form carbides when the amount of Carbon is sufficient. While carbon addition is beneficial for grain boundary ductility, the large carbides that form can adversely affect fatigue life (length greater than 0.005 mm) (Cetel A. D., Duhl D. N. 1988).

Cast superalloys microstructure is characterized by dendritic segregation, Fig. 1. Dendritic segregation is formed due to a significant chemical heterogeneity and secondary dendrite arm spacing (SDAS - factor) is very often evaluated as typical for cast superalloys. The colour metallography may help to identify carbide particles formed in inter-dendritic areas (Fig. 1).

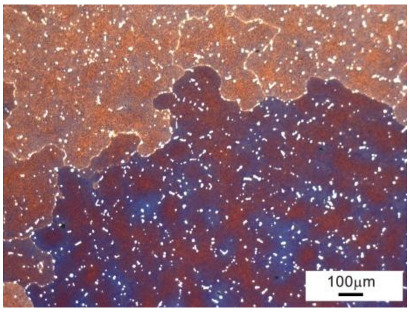


Fig. 1. A dendritic segregation in cast superalloys, ŽS6K; etch. Beraha III.

Microstructure of wrought superalloys consist of polyedric grains of various size, and depends on mechanical working. Due to mechanical history of microstructure, the deformation twins is presented in such a microstructure very often, Fig. 2.

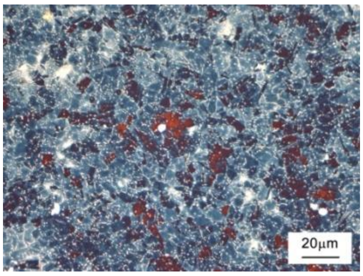


Fig. 2. A typical microstructure of wrought superalloys with polyedric grains and deformation twins, Alloy 718; etch. Beraha III.

SEM (Scanning Electron Microscopy) is employed for more detailed microstructure observation for a metallography process. At higher magnification it is possible evaluate morphology of γ'-phase and in some cases is possible to observe a γ/γ' eutectic cells formed especially in cast superalloys, Fig. 3.

From experimental fatigue data, S-N curves were plotted.

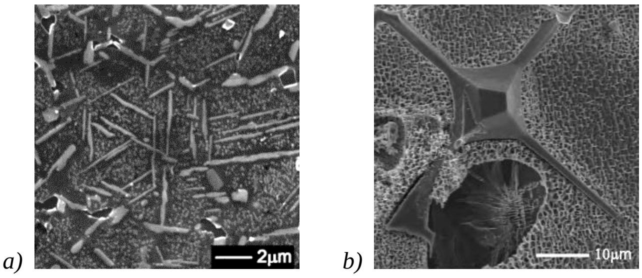


Fig. 3. SEM micrographs of a) alloy 718, electrolyte etching, CrO_3+H_2O (2V, 1s, 90 mA/cm^2) to reveal γ'-phase and δ-phase after annealing at 800 °C/72 hrs b) γ/γ' eutectic cells, morphology of $M_{23}C_6$ carbides is also visible, ŽS6K superalloy; etch. Marble.

Results can be seen at Fig. 4 where Fig. 4a reports fatigue lifetime curve for push-pull loading and Fig. 4b reports fatigue lifetime curve for three point bending loading (Belan J. et al. 2014, Uhríčik M. et. al. 2016, Kopas P. et. al. 2014, Bokůvka O. et. al. 2012). It is obvious that loading mechanism has an important influence on fatigue lifetime limit σ_c. While at push-pull high frequency is σ_c = 330 MPa at $N_f = 1.68\ .10^8$ at three point flexure low frequency loading was σ_c = 700 MPa (almost twice as high as push-pull loading) at N_f = 2.01 .10^7. SEM observation of fatigue fracture surfaces is shown at Fig. 5.

At higher volume of loading ~~a~~ multiple initiation sites can be seen in both cases, push-pull as well as three point flexure loading (Fig. 5a,b). After fatigue crack initiation, the major crack propagates at perpendicular direction to main loading and creates the so called "striation". It was characterised by transcrystalline mechanism of propagation (Fig. 5c, d).

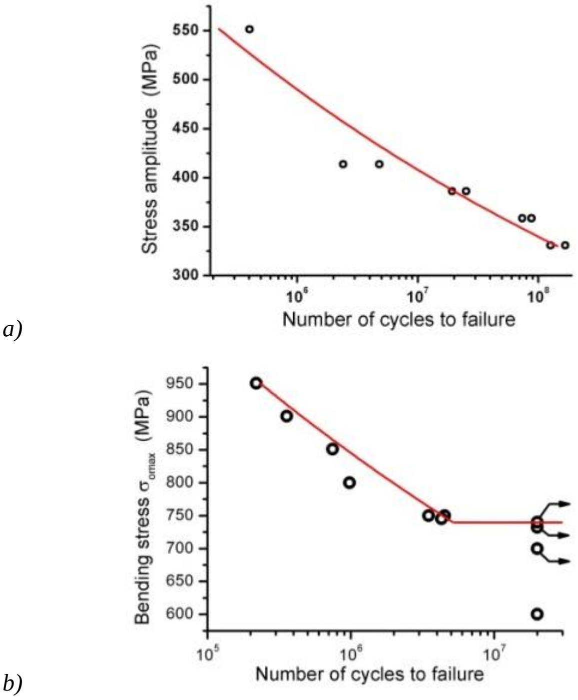
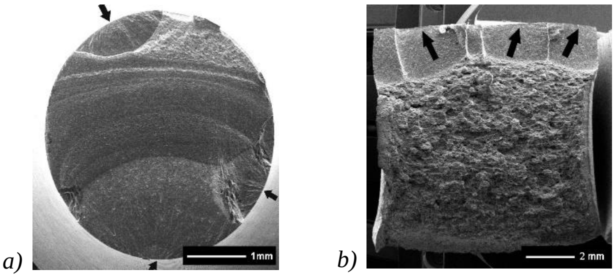


Fig. 4. Fatigue lifetime S-N curves for a) push-pull, high frequency loading, and b) three point bending, low frequency loading.



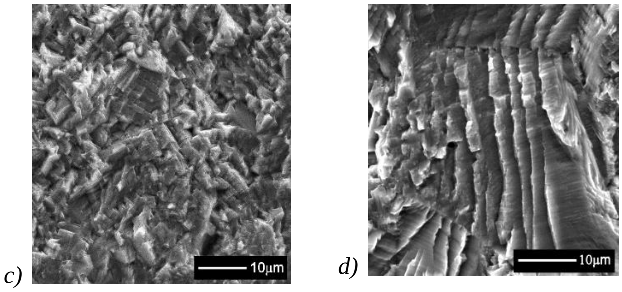


Fig. 5. SEM observation of fracture surfaces after fatigue tests; a-b) initiation site at higher cyclic loading, note three initiation points for push-pull (a) and three point flexure (b); (c-d) stable fatigue crack propagation characterised by striation (c is for push-pull; d for three point flexure).

4. Conclusions

The metallography of cast and wrought Ni-base superalloys is discussed in this paper. After preparation metallography specimens are etched with various reagent (chemically or electrolytic) which provide different results. Also colour etching for better identification of structural characteristics is successfully used at superalloys.

Metallography of cast superalloys has its own specifications because of significant dendritic segregation and its main task is to reveal dendritic structure of cast superalloys to help calculate and evaluate SDAS-factor.

On the other hand, wrought superalloys are prepared with the aim to reveal grain boundary and grain size. Its further objective is to evaluate deformation history of wrought superalloys such as the presence of deformation twins in structure.

No matter whether cast or wrought superalloys are taken into consideration, SEM is powerful tool to evaluate morphology of γ'-phase, and observe morphology of primary and secondary carbides. For example, coarse γ'-phase means decreasing of mechanical properties due to easier slip of dislocations. To achieve this aim are deep etching or electrolyte etching employed to reveal microstructure.

All described metallography techniques were employed to evaluate samples where the fatigue tests were applied. Metallography is a powerful tool for fatigue failure mechanism description and provides information how microstructure is related to mechanical properties. SEM observation reveals e. g. initiation sites of fatigue cracks, mechanism of crack propagation (striations and transcrystalline ductile mechanism of propagation), to static ductile dimple failure.

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Literature

1. RADAVICH J. F. 1997. Electron Metallography of Alloy 718, in: E.A. Loria (Ed.), Superalloys 7X3, 625, 706 and Various Derivatives, The Minerals, Metals & Materials Society, pp. 17-26.
2. VANDER VOORT G., MANILOVA E. P. 2004. In: Microscopy and Microanalysis, S02 (2004), 690-691.
3. Information on http://www.georgevandervoort.com /metallography/general/nickel-alloys/20001279-metallography-of-superalloys.html
4. RADAVICH J. F. 1988. In: Journal of Metals, 40 (1988), 42.
5. SKOČOVSKÝ P., PODRÁBSKY T. 2001. Colour metallography of ferrous alloys, EDIS, Žilina.
6. BELAN J. 2012. Study of advanced materials for aircraft jet engines using quantitative metallography, in: R.K. Agarwal (Ed.), Recent Advances in Aircraft Technology, IN-TECH - Open Access publisher of Scientific Books and Journals, Croatia, 2012, pp. 49-74.
7. DONACHIE M. J., DONACHIE S. J. 2002. Superalloys – A Technical Guide, 2nd edition, ASM International, USA.
8. PINEAU A., ANTOLOVICH S. D. 2009. In: Engineering Failure Analysis 16 (2009) 2668-2697.
9. CETEL A. D., DUHL D. N. 1988. In: 2nd International SAMPE Metals Conference, USA, pp. 37 – 48.
10. BELAN J., HURTALOVÁ L., VAŠKO A., TILLOVÁ E. 2014. In: Manufacturing technology: journal for science, research and production, 14 (2014) 262-267.
11. UHRÍČIK M., SAPIETA M., STANKOVIČOVÁ Z., PALČEK P., ORAVCOVÁ M. 2016. In: Materials Today: Proceedings (3) 2016, 1189-1194
12. KOPAS P., VAŠKO M., HANDRIK M. In: Applied Mechanics and Materials (474) 2014 285 – 290
13. BOKŮVKA O. ET AL. 2012. Annals of Faculty Engineering Hunedoara – Int. Journal of Engineering, Vol. X, No. 2.