

THE INFLUENCE OF APPLIED HEAT-TREATMENT ON IN 718 FATIGUE LIFE AT THREE POINT FLEXURAL BENDING

Received – Priljeno: 2016-04-25

Accepted – Prihvaćeno: 2016-10-10

Preliminary Note – Prethodno priopćenje

The Inconel alloy 718 is an iron-nickel based superalloy with a working temperature up to 650 °C. Presented phases such as γ'' (Ni_3Nb), γ' (Ni_3Al), and δ (delta – Ni_3Nb) are responsible for the alloy's unique properties. The δ – delta phase is profitable when situated at grain boundaries in small quantities due to increasing fatigue life. However, at temperatures close to 650 °C the γ'' transforms to δ – delta and causes a decrease in fatigue life. Heat-treatment (800 °C/ for 72 hours) and its influence on fatigue life are discussed in this paper. Fatigue tests were carried out at room temperature. After the tests we plotted the S-N curves for both stages. SEM (Scanning Electron Microscopy) fractography was carried out as well.

Key words: IN 718 alloy, heat-treatment, fatigue test, S-N curve, SEM fractography

INTRODUCTION

Mechanical properties of Ni-based superalloys heavily depend on the final microstructure, applied heat-treatment and of course on the stage (cast or wrought). It is a well-known fact about the positive influence of small grain size on tensile strength as well as fatigue lifetime below the limit temperature of 650 °C [1–5].

Heat treatment of such alloys is commonly reported with T-T-T diagrams (Time-Temperature-Transformation). The first T-T-T diagram for wrought alloy 718 was presented by Eiselstein in 1965 [6]. It is clear from all the published literature on alloy 718 that the beginning of precipitation and the type of the precipitate depend on the nature of the starting material, i.e. the degree of residual strain, the amount of delta produced by the solutioning step, and probably the most important is the degree of Nb segregation left after the processing cycles.

The current T-T-T study was undertaken to assess the phase reactions in uniform grain size billet material which was considered to be fully homogenized but still contains a certain amount of strain produced by forging. Figure 1 shows the T-T-T diagram plotted based on this study [7].

The strengthening phases in Alloy 718 are γ' (Ni_3Al) and mainly γ'' (Ni_3Nb), which is considered the main precipitation hardening phase responsible for the unique mechanical properties. The γ'' phase has a DO_{22} BCT crystal structure, while the γ' is a FCC ordered phase with a L1_2 crystal structure. The γ'' and γ' phases have unique morphologies which sometimes help to identify the phases. The γ' phase precipitates as a round particles whose size can be less than 200 Å, and continues to be round in shape when it coalesces at higher temperature.

The γ'' phase has more of a disk shape nature whose length is 5 to 6 times its thickness.

The delta phase found in alloy 718 is incoherent with the solid solution matrix γ and has an orthorhombic crystal structure. The delta phase (Ni_3Nb) is found mostly as plates growing on the (111) planes or nucleating on the grain boundaries, and is associated with a loss of strength in this alloy. The delta phase in the grain boundaries is used to control the grain size in wrought materials and seems to be also important for notch ductility [7].

The following study deals with the influence of re-heating of IN 718 alloy at temperature 800 °C for 72 hours. The chosen temperature and holding time correspond to the current T – T – T diagram, Figure 1. The temperature and holding time are situated in the area where a sufficient amount of Nb may cause coarsening of both the strengthening phases γ'' as well as the γ' , and becomes more visible when SEM techniques of observing are used. However, an increased amount of especially delta phase may cause significant deterioration of mechanical properties. The next part of the study fo-

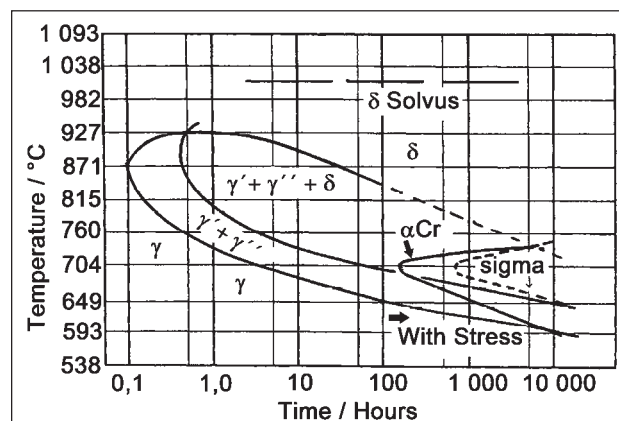


Figure 1 T-T-T diagram according to work of and Armida Oradei-Basile and J. F. Radavich [7]

cuses on the applied heat treatment influence on low-frequency fatigue lifetime. After a three-point flexural fatigue test at room temperature we plotted a S-N curve and compared it with the non-heat treated stage (the stage as received from BIBUS Company).

MATERIAL AND EXPERIMENTAL METHODS

The experimental material bars used in this study is a Ni-based INCONEL 718 superalloy.

The material was heat treated, according to the suppliers – BIBUS Ltd. (CZ) – of the material sheet, 980 °C/ 1 hrs. AC + heating at 720 °C/ 8 hrs. followed FC (50 °C per hour) to temp. 620 °C, holding time 8 hrs. and air cooled. The achieved mechanical properties of the material with grain size ASTM 12 are shown in Table 2.

The experimental material of the above-mentioned chemical composition was cut into simple blocky samples with dimensions (bxhxl) 11 mm x 10 mm x 56 mm.

Table 1 **Chemical composition of IN 718 (wt. %)**

C	Ni + Co	Mn	Ti	Al	B	Co
0,026	53,46	0,07	0,96	0,57	0,004	0,14
Ta	Nb + Ta	Cr	Cu	Mo	Nb	Ni
<0,01	5,31	19,31	0,03	2,99	5,30	53,32

*Fe - balance

The three-point flexural fatigue tests, Figure 2, were carried out on the testing machine ZWICK/ROELL Amsler 150 HFP 5100 at room temperature with static pre-load $F_{static} = -15$ kN and dynamic force $F_{dynamic}$ varying from 6,31 kN up to 12,8 kN. The value of $2,10^7$ cycles was set as reference and when specimen reached this value without break, the so-called runout, that bending stress was considered as fatigue lifetime limit. The frequency of fatigue tests was approximately $f = 150$ Hz.

Equation (1) was used for the calculation of the maximum bending stress:

$$\sigma_{Omax} = \frac{3 \cdot F \cdot L}{2 \cdot b \cdot h^2} / \text{MPa} \quad (1)$$

where σ_{Omax} is the maximum bending stress / MPa, F is the dynamic load / N, L is the distance of supports / mm, b and h are the specimens width and height / mm.

We also carried out fractographic analysis of the broken specimens, using scanning electron microscope TESCAN Vega II LMU. The fractographic analysis was done in order to describe micro-mechanisms of fatigue crack initiation, fatigue crack propagation (FCP) and final static fracture of specimens.

RESULTS AND DISCUSSION

The superalloy consists of the austenitic FCC matrix phase γ (solid solution of such elements as Cr, Fe, and Co in Ni) plus a variety of secondary phases. Secondary phases of value in controlling properties are the fcc car-

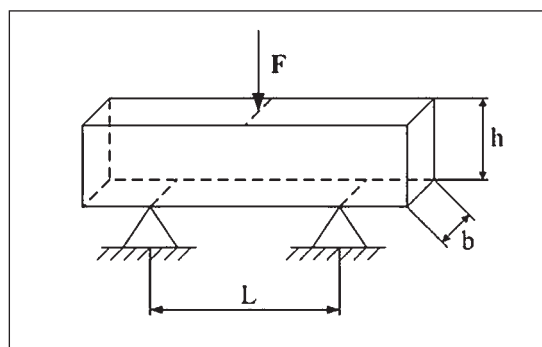


Figure 2 Schematic drawing of the three-point flexural fatigue test setting.

Table 2 **Mechanical properties of IN 718**

	Temperature / °C	
	20	649
Rp 0,2 / MPa	1 213	986
Rm / MPa	1 549	1 123
A / %	21,3	22,6
Z / %	33,3	68,0
HB 10/3000	429	-
$\sigma_{T/649}$ / MPa	-	689
Rupture life / hrs.	-	26,8
A / % creep	-	45,7

bides MC, $M_{23}C_6$, M_6C , and M_7C_3 (rare) in virtually all superalloy types; gamma prime (γ') fcc ordered $Ni_3(Al,Ti)$; gamma double prime (γ'') bct ordered Ni_3Nb ; and the delta (δ) orthorhombic Ni_3Nb intermetallic compounds in iron-nickel-based superalloys. The γ' , γ'' , and η phases also are known as geometrically close-packed (GCP) phases.

The tested alloy microstructure at the starting stage is presented in Figure 3a, and Figure 3b shows it after applied heat-treatment 800°C/72 hrs. Generally, the microstructure consists of very fine grain sizes according to ASTM = 12 (length of grains is around 10 μm), and various forms of segregated δ phase (light gray particles). The δ phase is presented mainly at grain boundary in a needle-like shape and in form of fine blocks inside the grains. However, the needle-like morphology of the δ phase can be found also inside the grains. The presence of γ'' or γ' was hard to prove and observe due to the low temperature (phases γ'' and γ' became more obvious after exposure to higher temperatures – because of its

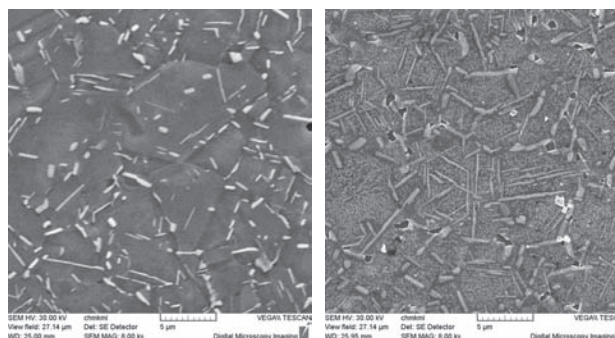


Figure 3 Microstructure of wrought INCONEL alloy 718 as seen in SEM, Kalling's etch

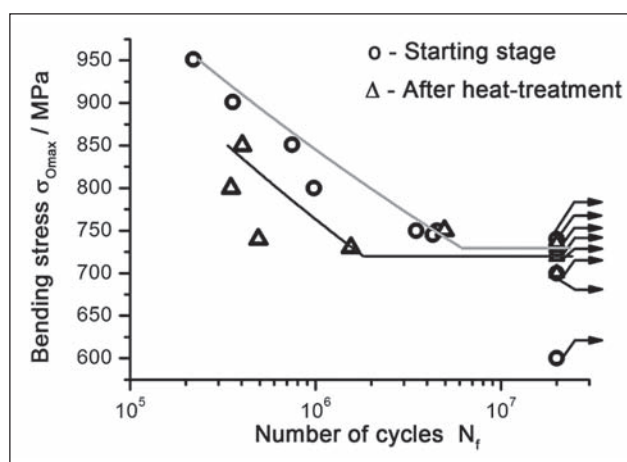


Figure 4 Fatigue test S-N curve of Inconel Alloy 718 at $f \approx 150\text{Hz}$, room temperature

growing) and insufficient magnification of SEM observation. After application of heat-treatment the secondary strengthening phase γ' became more obvious because of temperature and δ phase grew as well. According to reference [8], mechanical properties should deteriorate due to the δ phase presence at grain boundaries and its growth.

For low frequency three-point bending fatigue testing we used specimens with a simple blocky shape. Loading and testing procedures were the same for specimens at the starting stage as well as for heat-treated specimens. Figure 4 shows the S-N curve of IN718 obtained from the three-point bending fatigue test at room temperature.

The obtained results were approximated with equation (2), which is a Basquin formula for S-N presenta-

tion and approximation for the starting stage tests, and equation (3), again the Basquin formula but for the heat-treated stage. This approach was also used for different materials [9].

$$\sigma_a = 2425,1 \cdot N_f^{-0,078} \quad (2)$$

$$\sigma_a = 1202,4 \cdot N_f^{-0,032} \quad (3)$$

where $\sigma'_f = 2425,1$ (or 1202,4 for the heat-treated stage) is a coefficient of fatigue strength and $-0,078$ (or $-0,032$ for the heat-treated stage) = b is the lifetime curve exponent.

The measured S-N curve clearly shows that the fatigue life increases with the decreasing stress amplitude and the S-N curve appears to continuously decline as the life extends.

Comparison of the S-N curves for the starting stage and after applied heat-treatment shows two interesting facts. Firstly, there is a downtrend of samples at the starting stage S-N curve that is steeper compared to the heat-treated samples S-N curve. Secondly, the achieved fatigue limit of $2 \cdot 10^7$ cycles corresponds with the load $\sigma_{Omax} = 735$ MPa for the starting stage and $\sigma_{Omax} = 725$ MPa for the heat-treated stage. The truth is that the difference between the measured values should be higher, due to the metallographic observation with coarse γ' and more δ phase presence at the heat-treated stage. The reason is that the set temperature and holding time 72 of hours do not have such influence on lower fatigue lifetime of load amplitude at three-point bending.

Figure 5 shows an overall view of the fracture surfaces at different stress levels as well as different conditions (starting stage or heat-treated stage). Initiation sites are marked by an arrow. As a confirmation of the facts published in [10], as shown in Figure 5a, fatigue crack

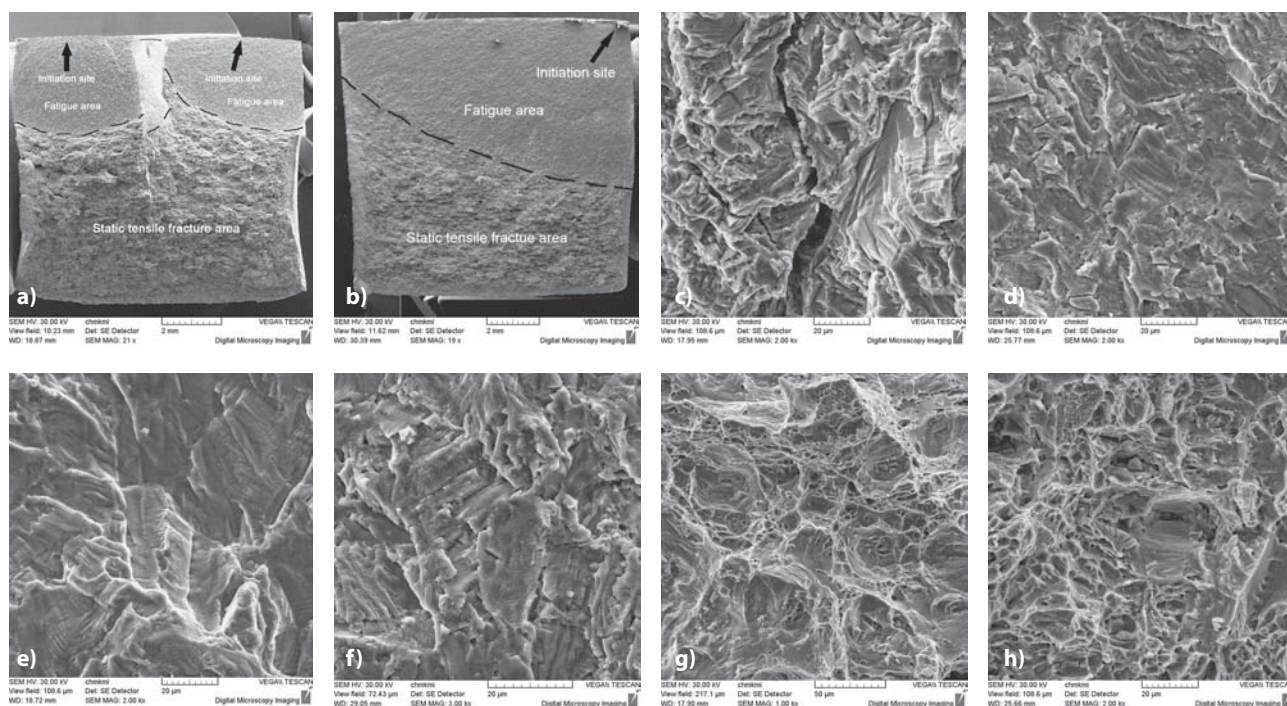


Figure 5 Overview of the surfaces in the Low Frequency Fatigue (LFF) regime, SEM. a) $\sigma_{Omax} = 851$ MPa, $N_f = 7,49 \times 10^5$ cycles; b) $\sigma_{Omax} = 850$ MPa, $N_f = 4,09 \times 10^5$ cycles; c) $\sigma_{Omax} = 851$ MPa, $N_f = 7,49 \times 10^5$ cycles d) $\sigma_{Omax} = 750$ MPa, $N_f = 4,96 \times 10^6$ cycles; e) $\sigma_{Omax} = 800$ MPa, $N_f = 9,79 \times 10^5$ cycles – very fine striations; and f) $\sigma_{Omax} = 740$ MPa, $N_f = 4,92 \times 10^6$ cycles – very fine striations; g) and h) static fracture area, SEM

initiated from multiple initiation sites at high stress levels. This fact is valid only in the case of the starting stage specimens. Specimens after applied heat-treatment have only a single initiation site, regardless of loading - Figure 5b. After initiation, fatigue crack propagates in a transcrystalline manner with typical striations, and indicates the stable crack propagation as shown in Figures 5c, e – the starting stage and 5d, f – the heat-treated stage. Figures 5e and 5f show very fine striations.

Major cracks occur in both cases (Figure 5c – the starting stage, and Figure 5d – the heat-treated stage). They are perpendicular to the main direction of crack propagation. Figure 5g – the starting stage, and Figure 5h show a classical transcrystalline ductile dimpled fracture mechanism in the tensile final fatigue region.

CONCLUSIONS

Nickel-based INCONEL 718 superalloy was subjected to low frequency three-point bending fatigue test up to $\approx 2 \cdot 10^7$ cycles at room temperature. There were two set of specimens; the first ones in the starting condition as supplied by BIBUS Ltd. (CZ), the second ones after applied heat-treatment at 800 °C/ 72 hrs. with the aim to increase the level of the δ phase and produce a coarse γ' phase. This heat-treatment should lead to deteriorated mechanical properties and shortened fatigue lifetime as well. After the experiments, the conclusions are as follows:

- The alloy microstructure consists of lenticular and needle-like particles of a stable δ (Ni_3Nb) orthorhombic phase, and of light gray blocks of mostly primary carbides MC created by Ti and Nb.
- The S-N curve in LFF of alloy 718 at the starting stage and after applied heat-treatment has been measured. The S-N curve downtrend of specimens at the starting stage was steeper compared to the heat-treated stage. Fatigue limit σ_{Omax} differs only slightly (735 MPa compared to 725 MPa at the heat-treated stage). The applied heat-treatment does not significantly influence the fatigue lifetime at three-point bending loading.
- Fractographic analysis shows that, at higher stress level, a fatigue crack initiates from multiple sites. At the heat-treated specimens the fatigue crack initiates

from a single site. After initiation the crack propagates in a typical transcrystalline manner.

Acknowledgements

The authors acknowledge the financial support of the project VEGA No 1/0533/15 and KEGA No 044ŽU-4/2014.

REFERENCES

- [1] R. L. Saha, K. Gopinath, K. K. Sharma, M. Srinivas, Low cycle fatigue behaviour of alloy 718 disc forging at elevated temperatures, *Superalloys 718, 625, 706 and derivatives*, (2001), ed. by E. E. Loria, TMS.
- [2] W. L. Mills and C. M. Brown, Fatigue fracture surface morphology for alloy 718, *Superalloys 718, 625, 706 and derivatives*, (2001), ed. by E. E. Loria, TMS.
- [3] J. Warren, D.Y. Wei, The cyclic fatigue behaviour of direct aged 718 at 149, 315, 454 and 538 °C, *Materials Science and Engineering A* 428 (2006), 106-115.
- [4] S. P. Lynch, T. C. Radtke, B. J. Wicks and R. T. Byrnes, Fatigue crack growth in nickel-based superalloys at 500-700 °C. II: Direct aged alloy 718, *Fatigue Fract. Engng Mater. Struct.* 17 (1994), 3, 313-325.
- [5] J. Belan, High frequency fatigue test of IN 718 alloy – Microstructure and fractography evaluation, *Metalurgija*, 54 (2015) 1, 59-62.
- [6] H. L. Eiselstein, Metallurgy of a Columbium-Hardened Nickel-Chromium-Iron Alloy, ASTM Special Technical Publication 369 (1965), 65-79.
- [7] A. Oradei-Basile, J. F. Radavich, A current T-T-T diagram for wrought alloy 718, *Superalloys 718, 625 and Various Derivatives*, ed. By E., A. Loria, TMMMS, (1991), 325-335.
- [8] D. G. L. Leo Prakash, M. J. Walsh, D. Maclachlan, A. M. Korsunsky, Crack growth micro-mechanisms in the IN718 alloy under the combined influence of fatigue, creep and oxidation, *International Journal of Fatigue* 31(2009)11-12, 1966-1977.
- [9] L. Trško, O. Bokůvka, M. Guagliano, F. Nový, M. Hazlinger, 30th Danubia-Adria Symposium on Advances in Experimental Mechanics, Croatian Society of Mechanics 2013, pp. 131-132
- [10] X. F. Ma, Z. Duan, H. J. Shi, R. Murai, E. Yanagisawa, Fatigue and fracture behavior of nickel-based superalloy Inconel 718 up to the very high cycle regime, *Journal of Zhejiang University-SCIENCE A (Applied Physics & Engineering)* 11 (2010) 10, 727-737

Note: The responsible translator for the English language is Mgr. Jana Súkeníková, Žilina, Slovak Republic