The High-Temperature Loading Influence on Orthorhombic Ni_3Nb DO_a δ - Phase Formation and its Effect on Fatigue Lifetime in Alloy 718

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The orthorhombic DO_a Ni_3Nb delta phase is naturally presented in microstructure of cast or wrought Ni – Cr – Fe base alloy 718. It is a reason of chemical composition heterogeneity. For delta phase formation is a minimum Nb content about 8% needed and temperature above 650°C and higher. A typical place, where delta phase is situated are grain boundaries in alloy 718, where its role is discutable. Some references saying about lowering the mechanical properties when delta phase is segregated at grain boundary. To approve or disprove of this fact, the wrought alloy 718 with ASTM 12 grain size was subjected to high-temperature loading at push – pull fatigue at low cycle and low frequency (LCLF) and obtained results were compared to results from very high cycle and frequency (VHCF) push – pull loading datas obtained in previous experiments done at room temperature. To support results about influence of delta phase formation an observation on light microscopy and SEM was made too. To increase and make easier identification of delta phase, the techniques of polarised light (DIC - Differential Interferential Contrast) and dark-field (DF) observation were used too.

Keywords: High-temperature loading, Delta phase formation, Microstructure analysis, Dark-field optical microscopy, SEM observation

1 Introduction

It has been decades what designers are using and enjoying the benefits of age-hardenable Ni – Cr – Fe superalloy IN 718 at turbine construction. The outstanding properties of this alloy, such are corrosion resistence at high temperatures, good weldability, tensile, fatigue, and creep properties results from its unique microstructure. IN718 alloy consists of a FCC γ-matrix with δ phase, MC carbides and a high volume fraction of embedded γ' and γ" precipitates [1-3]. Contrarily to most of the superalloys, it is the coherent body-centered tetragonal (BCT) γ" (Ni_3Nb) precipitates that is the main hardening phase; the face-centered cubic (FCC) structure γ' Ni_3(Al, Ti) precipitates only brings a slight additional strength to the alloy [1, 3, 4]. The γ" is actually a metastable phase. It transforms into a stable but incoherent δ phase (Ni_3Nb) with orthorhombic structure if the alloys is exposed to long ageing time or to temperatures higher than 650 °C or even during service. The δ phase precipitates at grain and twin boundaries or in an intragranular manner with a plate shaped or globular morphology [5, 6]. Since γ" and δ have the same composition, the growth of the δ phase occurs to the detriment of the γ" phase [7].

As was mentioned above, the IN 718 alloy is age-hardenable thanks to various Nb, Al, and Ti elements content. From that point of view the aditional heat-tretment after annealing is very important. Several authors have detailed the precipitation ranges for the main phases of IN718 alloy and a few TTT diagrams (Temperature-Time-Transformation) have been drawn [8, 9, 10, 11]. According to many authors, the γ' and γ" phases precipitate between 600 °C and 900 °C, whereas the highest level of δ precipitation is found around 900 °C [6, 7, 10]. The knowledge gained on the precipitation of the metastable and the equilibrium phases (γ" and δ) has been skillfully adopted by industry to decide the standard heat treatment procedure to produce this alloy in the proper age hardenable condition. The procedure involves solution treatment at 954 °C followed by ageing at 720 °C and 620 °C for specific periods of time. Heat treatment at higher temperature produces blocky δ particles, mainly at grain boundaries, to stabilize the grains, while heat treatments at lower temperatures are given to precipitate γ" particles in the matrix to strengthen the alloy. Heat treatments at temperatures lower than 954 °C has been employed to generate a microstructure (consisting of high volume fraction of uniformly dispersed fine δ particles) that is used during forging operations to produce fine grain grades of IN 718 alloy. At these high temperatures, the δ particles have been reported to have needle, plate as well as globular morphologies. Similarly, any abnormal rise in temperature or stress during service can also result in complete conversion of γ" precipitates to δ particles [12]. It is due to the thermo-dynamic proces, when γ" disolves, it releases alloying elements such Al, Ti and Nb and this leads to formation of δ and γ' phases according to precipitation sequence γ" (Ni_3(Nb, Al, Ti)) → δ (Ni_3Nb) + γ' (Ni_3(Al, Ti)).

A lot of researches were done about metastable γ" phase and its influence at various temperatures on mechanical properties. But the other very common phase, the δ phase, stays on background of interest. Information about it influence on mechanical properties is contrary. According to references [7, 12, 13] the δ phase has an orthorhombic D0a structure with Ni_3Nb stoichiometry and its unit cell is illustrated in Fig. 1. The orientation relation between the D0a structure and FCC matrix as reported in

the literature can be expressed as $\{111\}_{FCC} \parallel (010)_{D0_a}$ and $\langle 1\bar{1}0\rangle_{FCC} \parallel [100]_{D0_a}$. The morphology of the δ phase is not known to allow any significant contribution to the hardening of IN 718 alloy. It has also been reported that the presence of the δ phase decreases yield and tensile strengths but has a beneficial effect on the rupture ductility [7, 14]. Similar conclusion was drawn for creep properties [15]. However, very few studies report the possible relationship between the δ phase content and the formability of the material at room temperature. Only Valle et al. [13] studied the influence of the small amount of δ-phase volume fraction on the mechanical properties such as yield and tensile strengths, elongation, and hardness. They have shown that 0.2–1.5% δ phase do not influence the mechanical properties of aged samples, but yield strength, ultimate tensile strength, elongation, and hardness is influenced by grain size, Fig. 2.

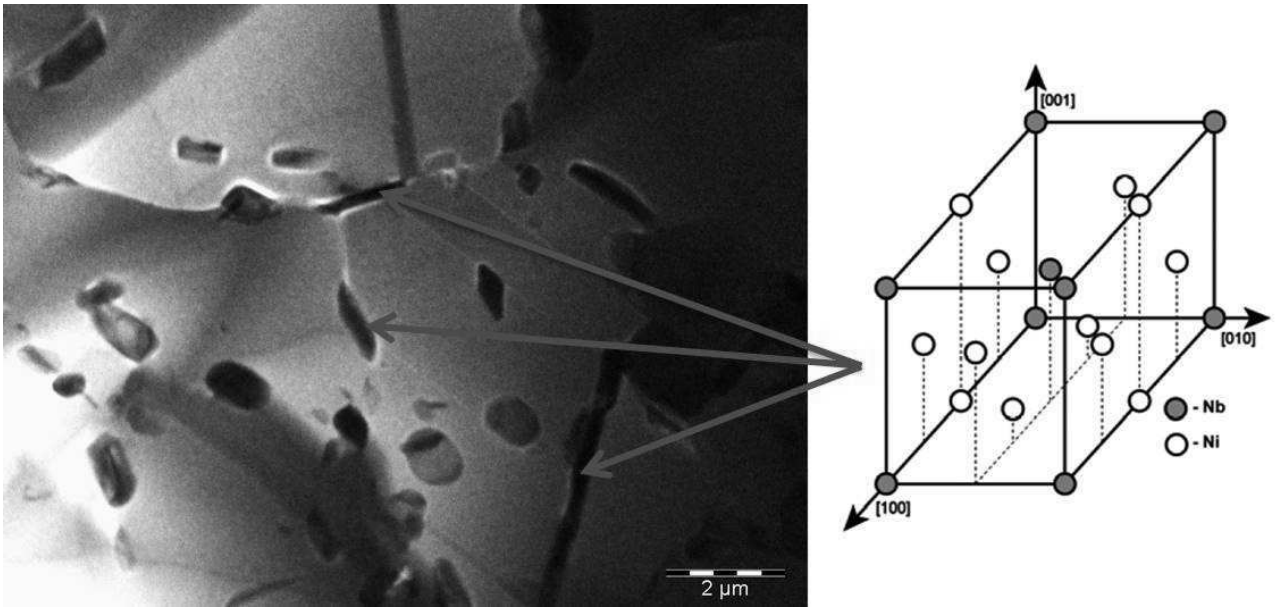

Fig. 1 *TEM micrograph of initial state of IN 718 alloy (left) and various types of δ phases with $D0_a$ lattice (right)*

Sundararaman et al. [6] shows that the formation of large amounts of coarse δ plates degrades the strength of the superalloys hardened by γ'' precipitates due to the corresponding depletion of the γ'' phase. No study has been conducted for higher amount of δ precipitates to confirm the general assumption that the presence of δ phase precipitation in IN 718 induced a ductility improvement in the material or realtion between the δ phase content and fatigue properties at high temperatures and how the combination of high temperature and mechanical loading influences the δ phase formation.

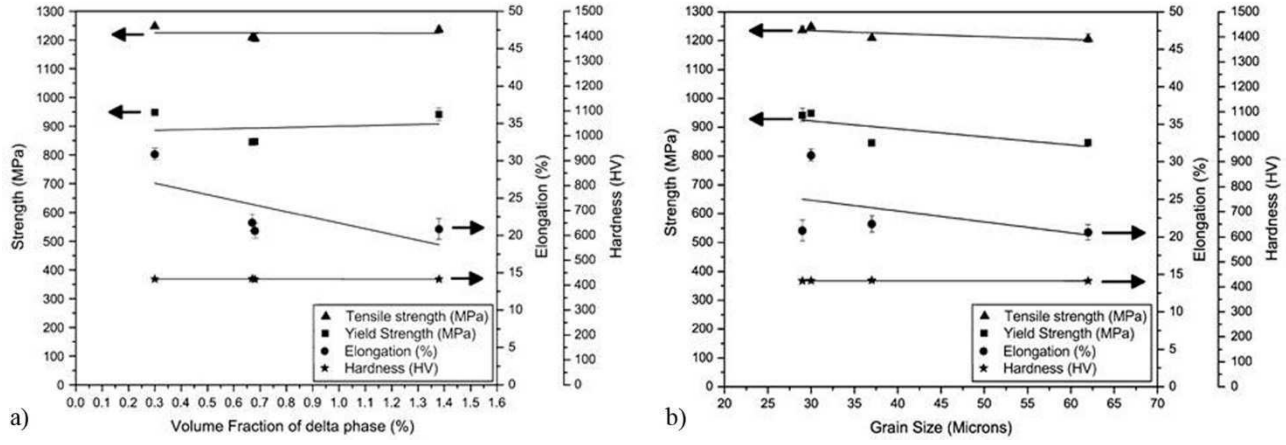

Fig. 2 *The influence of δ phase volume fraction (a) and the grain size (b) on ultimate tensile strength, yield strength, elongation, and hardness [13]*

The aim of this study is to determine how the δ phase formation affected the fatigue properties if the IN 718 alloy was subjected to push-pull symmetrical fatigue loading at room temperature (VHCF) and at temperature 700°C (LCLF).

2 Experimental material and methods

For experiments Ni – Cr – Fe IN 718 alloy was used. The alloy was supplied by BIBUS Metals AG, Brno, CZ

as cold drawn, solution and precipitation treated bar with 12 mm of diameter. The bar was vacuum induction melted and vacuum arc remelted with conditions as follows: at 980 °C/1 hr. and air cooled then at 720 °C/8 hrs. aged and cooled down with speed 50 °C/hr. to 620 °C held for 8 hrs. and then air cooled. The average grain size was ASTM 12 (what corresponds from 4.2 to 5.9μm of grain size). The chemical composition was obtained by spark spectrometer SpectroMAXx and is in Tab. 1.

Tab. 1 *IN 718 chemical composition in weight % from SpectroMAXx*

C	**Si**	**Mn**	**P**	**S**	**Cr**	**Fe**	**Mo**	**V**	**Cu**
0.0428	0.158	0.0742	<0.001	0.0013	22.3	16.45	2.82	0.0461	0.068
W	**Co**	**Nb**	**Al**	**Ti**	**Zr**	**Sn**	**Ta**	**B**	**Ni**
0.102	0.145	4.33	0.51	0.835	0.0224	<0.01	0.0134	0.314	52.0

The specimens for fatigue tests were turned from bar. For high frequency push-pull loading fatigue test at room temperature the specimens have glass-hour shape, Fig. 3a, and for low frequency push-pull loading fatigue test at 700 °C theyhave the dimensions according to Zwick/Roell drawings, Fig. 3b.

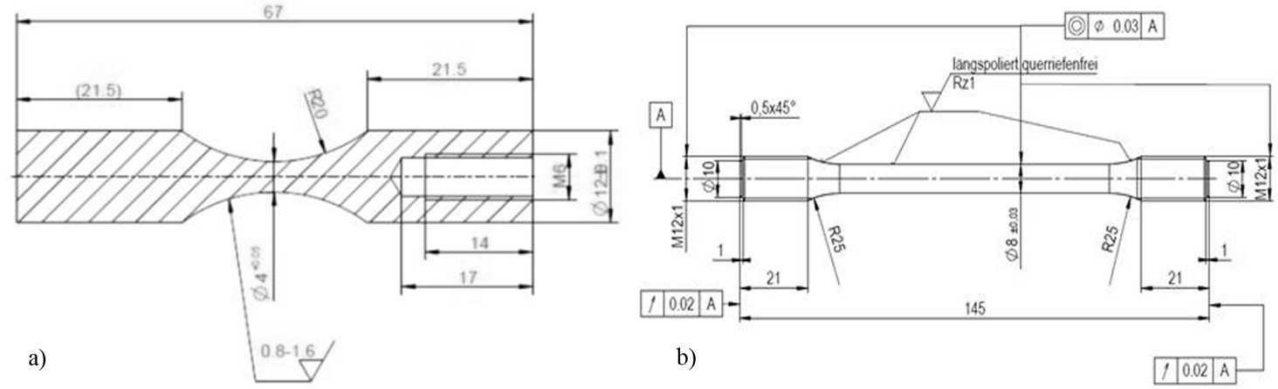

Fig. 3 *The shape and dimensions of fatigue test specimens used at room temperature high frequency loading (a) and for low frequency loading at 700 °C (b)*

The high frequency push-pull fatigue test at room temperature was carried out on experimental fatigue machine KAUP – ŽU with loading frequency 20 kHz, which was developed by Department of Materials Engineering, Faculty of Mechanical Engineering, University of Žilina in Slovak Republic. The loading was symetrical cycling with $\sigma_m = 0$ MPa and coefficient of cycle asymmetry was R = -1. The loading amplitude σ_a varied from 330 MPa to 551 MPa. The number of cycles 10^8 and higher was considered as fatigue limit – so called run-out.

The low frequency push-pull fatigue test at temperature of testing 700 °C was carried out on ZWICK / ROELL Amsler 150HPF 5100 resonance pulsator equipped by high temperature furnace with operating temperature up to 1200°C and loading frequency was approximately 64 Hz. The testing specimen was inserted into heating chamber and through ZWICK / ROELL testing software the temperature of 700 °C was set. When the furnace reached desired temperature the dwell time for heating specimen on whole crossection was 5 min. After that time the fatigue test started with parameters $\sigma_m = 0$ MPa, coefficient of cycle asymmetry was R = -1. The loading amplitude σ_a varied from 275 MPa to 452 MPa. The number of cycles $2x10^7$ and higher was considered as fatigue limit – run-out.

After fatigue test the specimens for optical microscopy (OM) and SEM observation were prepared. The preparation consisted of cutting the specimens in longitudinal direction of sample, perpendicular to fracture surface. After this operation the specimens were mounted into dentacryl, grinded and polished on Struers TegraPol – 15 with selected program on Struers TegraDoser – 5. The programm steps were as follows:

- SiC abrasive paper grain 220, lubricated by water – coarse grinding for 2 minutes,
- Largo with suspension DiaP. All / Lar. – fine grinding for 4 minutes,
- Dac with suspension DiaP. Dac – polishing for 4 minutes,
- fine polishing with OP – S for 2 minutes.

After preparation samples were rinsed in water and alcohol, dryied and etched by KALLINGS II. etchant reagent spread by cotton swab for 15 seconds.

The volume of soluted δ phase was observed in starting stage, as alloy was received, with optical microscope and SEM and TEM eventualy and than evaluated and compared after fatigue test. For observation of fatigued the same techniques as in starting stage were used. For an easier identification of δ phase the techniques of dark field (DF) observation (optical microscope) and SEM and TEM diffraction analysis were used. Of course, the secondary results are S-N curves, which were drawn after

fatigue tests and compared to each other For OM observation NEOPHOT 32 with dark-field filter was set on or off. SEM observation was done on TESCAN Vega II LMU equiped by EDX analysator. TEM observation was performed in Technical University of Košice, Institute of Materials.

3 Results

The microstructure of starting stage of IN 718 as received from supplier is shown in Fig. 4. It consists from austenitic grains of γ phase, what is solid solution of elements Cr, Fe in Ni lattice, primary carbides of NbC or TiC [16] type, and with annealing twings and the δ phase situated mostly at the grain boundaries, Fig. 4a. The γ" or γ' due to observation methods were not observed. As mentioned previously, the role of δ phase, when situated at grain boundaries, is to stabilize grain size at further mechanical treatment. The δ phase has morphology of plates or needles at grain boundaries or blocky shape when segregated intergranularly. The δ phase is incoherent with matrix, DO_a orthorhombic lattice parameters on diffraction pattern are a = 5.106 Å, b = 4.251 Å, and c = 4.556 Å, Fig. 4c. The length is 1.15μm and width is 0.51μm, Fig. 4b.

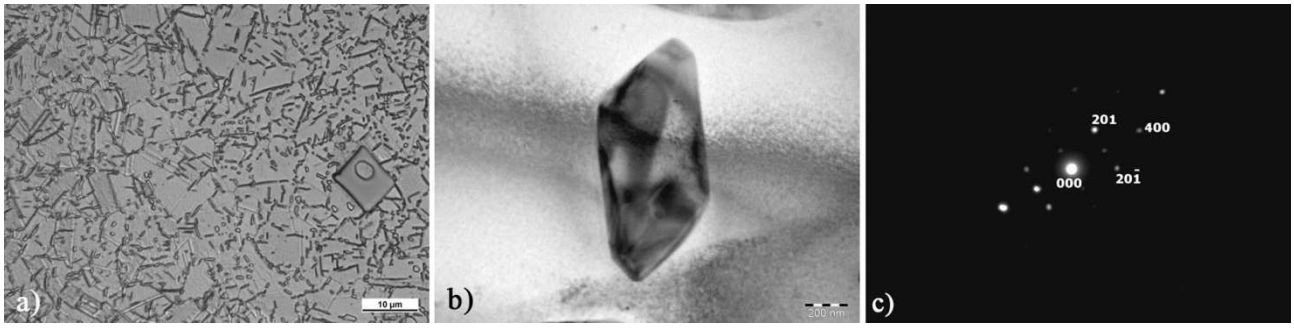

Fig. 4 *The microstructure of starting stage of IN 718 alloy, as received; a) the OM microstructure with δ phase situated at grains and twins boundary in plate or needle like shape, etch. Kallings II, b) the blocky shape δ phase segregated intergranularly observed with TEM and its diffraction patern (c)*

One randomly selected specimen after initiation stage analysis was used for analysis of the temperature influence on δ phase formation. The specimen was inserted into oven with pre-set temperature 700°C / 72 hrs. folowed by SEM and TEM observation. The microstructures are presented on Fig. 5.

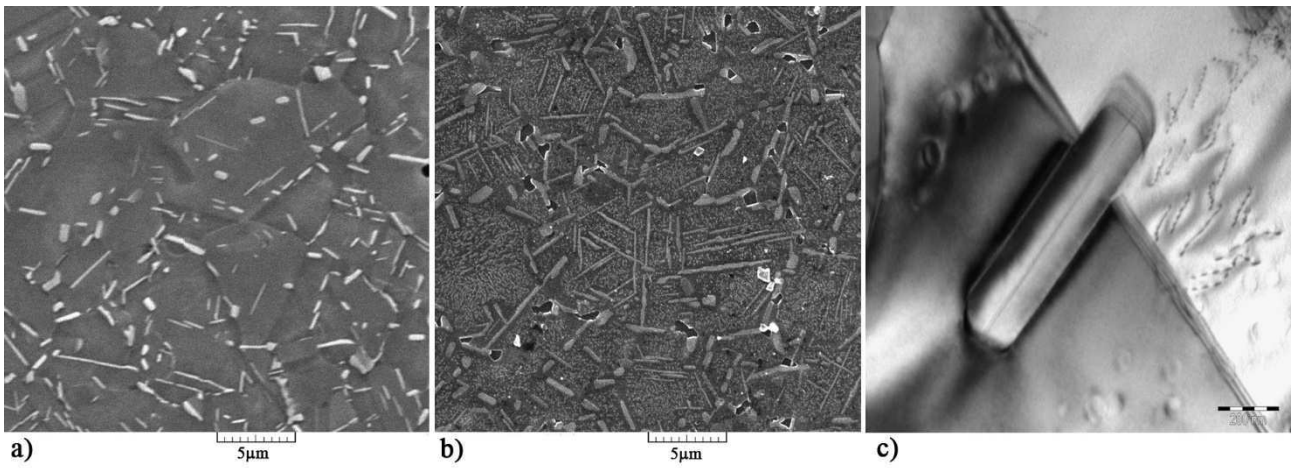

Fig. 5 *Comparision of starting stage with δ phase formation (a); after applied heat loading at 700 °C/72 hrs, δ phase formed in nedele like morphology at grain boundary and Widmanstätten morphology of δ phase segragated intergranularly (b) via SEM observation, and TEM micrograph with δ phase at twins boundary with dislocation lines around it*

Compared to starting stage of alloy (Fig. 5a) the δ phase became more coarse, forms in needele like morphology at grain boundary and as Widmanstätten needles intergranularly after aplied heating (Fig. 5b) [17-19]. The reason of that is used temperature where at this temperature range γ" phase became unstable and started to transform into δ phase. Chemical composition of both phases is the same, Ni_3Nb, differences are only in lattice of phases (γ" has BCT DO_{22} structure and δ has orthorhombic $D0_a$). The δ phase forms in higher volume when γ" phase releases Nb into solid solution. The second fact is that coherent γ' phase (presented in austenitic γ grains) gets coarse, Fig. 5b, and becomes more visible even at SEM observation. As a reason of applied temperature also dislocation lines occur (Fig. 5c) which create the dislocation front at twins boundaries and they are blocked by coarse γ' phase. Results about increased volume of δ phase after applied heat-treatment only and also after fatigue loading at 700 °C are shown in Tab. 2. The results were obtained by quantitative analysis with coherent testing grid. The used method and evaluation are described in reference

[20]. Measurement was done on SEM micrographs and result values are the medium from the ten measurements on every micrograph.

From measured results is obvious, that volume of δ phase fraction in starting stage is 39.5% and as was described above, the δ phase si situated at grain boundaries as needle like shape or in blocky shape intergranularly. With applied heat-treatment or mechanical loading at higher temperatures, volume fraction of δ phase rapidly increases in about 33% in the heat-treatment specimen and about 64.5% in the mechanically loaded specimen, Fig. 6.

Tab. 2 *Results of δ phase volume measurement by quantitative metallography method*

Alloy IN 718	**Volume of δ phase fraction V [%]**
Starting stage	39.5
After applied heating at 700°C/72 hrs.	52.5
After fatigue test at 700°C	65

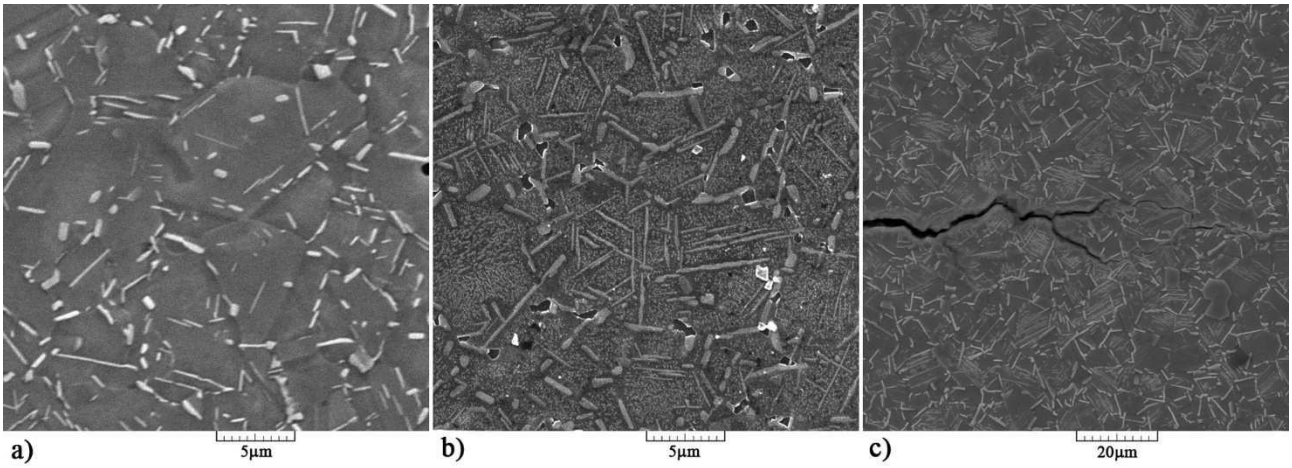

Fig. 6 *Comparison of δ phase volume fraction; a) starting stage, b) after applied heat-treatment 700 °C/72 hrs., and c) after fatigue test at 700 °C*

The coarse γ' phase can be easily viewed on Fig. 6b. This coarsening is always connected with loss of coherency to γ matrix and decreasing precipitation strength. In case of IN 718 alloy the main precipitation phase is γ'' so coarsening of γ' phase is not so big deal and does not significant impact on mechanical properties. The crack seen on Fig. 6c is the secondary fatigue crack; all fatigue cracks will be discussed later on in fatigue part.

While the microstructures after fatigue loading at 700 °C were analysed, the interesting fact was observed. Microstructure close to specimen centre was not so much "affected" by heat and looks almost the same as in starting stage. But close to the specimen surface where secondary fatigue crack has occurred, probably the combination of heat and mechanical loading have speed up the physical process of γ'' depletion and increased amount of δ phase in surrounding of that crack was observed, Fig. 7.

To help identify the δ phase, the DIC (Differential Interference Contrast, Fig. 7b, d) and DF (Dark Field, Fig. 7a, c) techniques on optical microscope were used. This observation method was used on etched samples. In the DF observation, the δ phase is white and really shiny while austenitic γ matrix remains black. This method is suitable to analyse the δ phase only and shows its distribution. When DIC used, the δ phase is colourfull (various colors) while austenitic matric γ is margenta or violet-brown. Compared of these two observation methods, the DF is better for δ phase identification but not so usefull when the crack occurs in the structure, because the crack is black as the γ matric and can be easy missed; when used DIC there is the problem to find the proper colour to separate the δ phase from γ matrix colour, in the other hand, the defferences between δ, γ'', and γ lattice are not so significant, so DIC did not works very well. Anyway, DIC is fine method to analyse fatigue crack propagation through material cross-section. From that point of view, the secondary fatigue crack propagates via transcrystalline and intercrystalline cleavage mechanism.

To fullfile the secondary goal, how the increased δ phase formation affect the fatigue properties, the fatigue push-pull test at room temperature and at 700 °C was done. The S – N curves as result of these tests are presented on Fig. 8. From obtained results is seen that the fatigue lifetime at room temperature test on specimens with δ phase volume fraction 39.5% is higher and corresponds to σ_a = 330 MPa at 1.68×10^8 cycles. At specimens fatigued at 700 °C with δ phase fraction volume 65%, σ_a = 275 MPa at 2.0×10^7 cycles. The stress amplitude σ_a value is about 20% lower compared to results at room temperature. However, when we take into account fact of run-out value and compare results at the same number of cycles, 2.0×10^7 this difference is even higher (40%, σ_a = 386 MPa at 1.9×10^7, room temperature approximation). Compared these two values the result is that increasing of δ phase fraction from 39.5% to 65% means decreasing of fatigue limit about 40% compared at the same number of cycles to failure.

Reason of lowering the fatigue lifetime is obviously increased volume of δ phase and of course the test temperature. Fatigue process is influenced with many factors, surface roughness, and stress amplitude, frequency of loading, microstructure and temperature as well. The influence of temperature, briefly, is mainly in oxide creation on the fracture surface, what obstructs the fatigue crack

closing mechanism while cycling. It completely changes the mechanism of fatigue crack propagation. The fatigue crack propagates via transcrystalline or intercrystalline mechanism, depends on grain orientation to applied stress and of course on the brittle or Widmanstätten needles phase presence inside of grains.

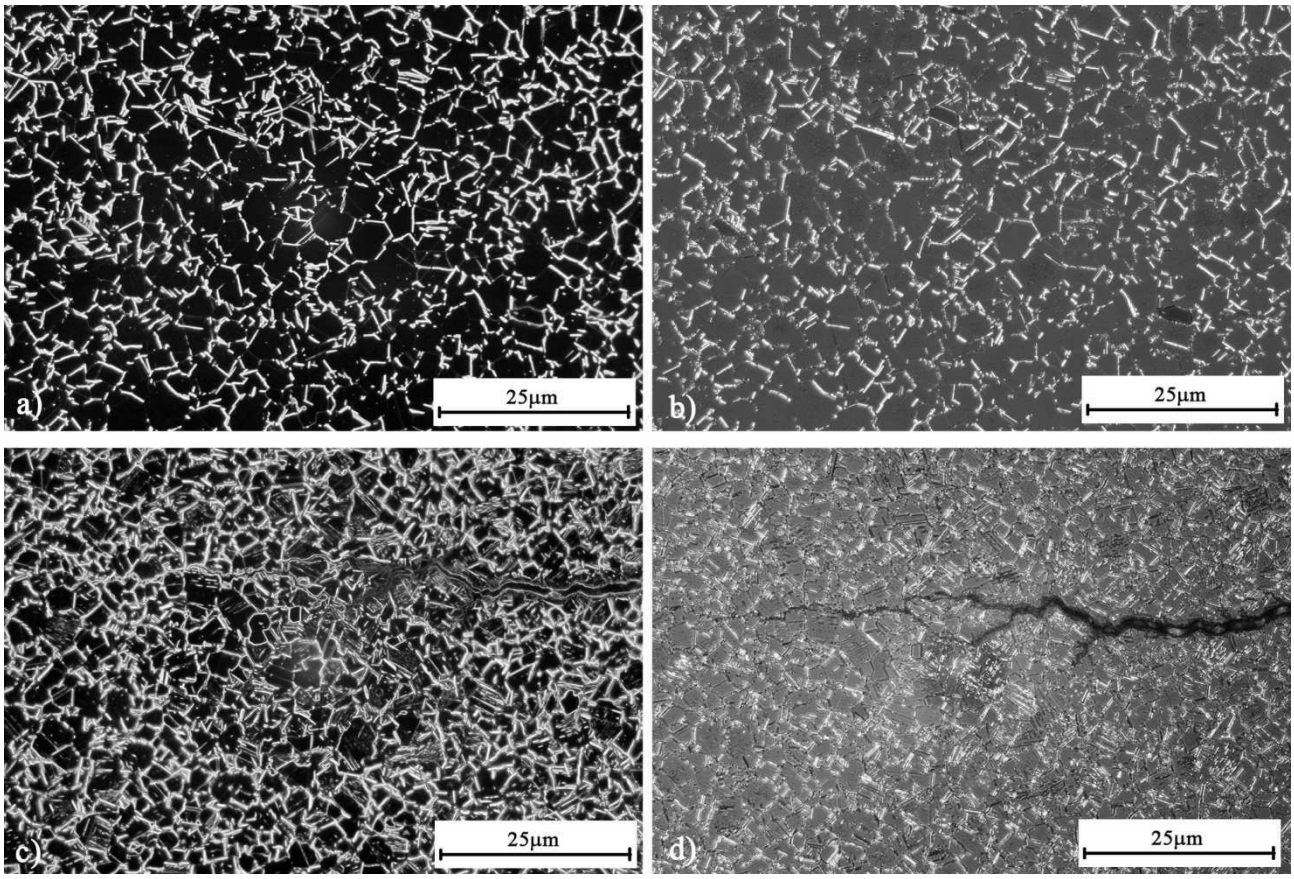

Fig. 7 *Various techniques of microstructure observation, DF (a, c) and DIC (b, d) to easier identify the δ phase*

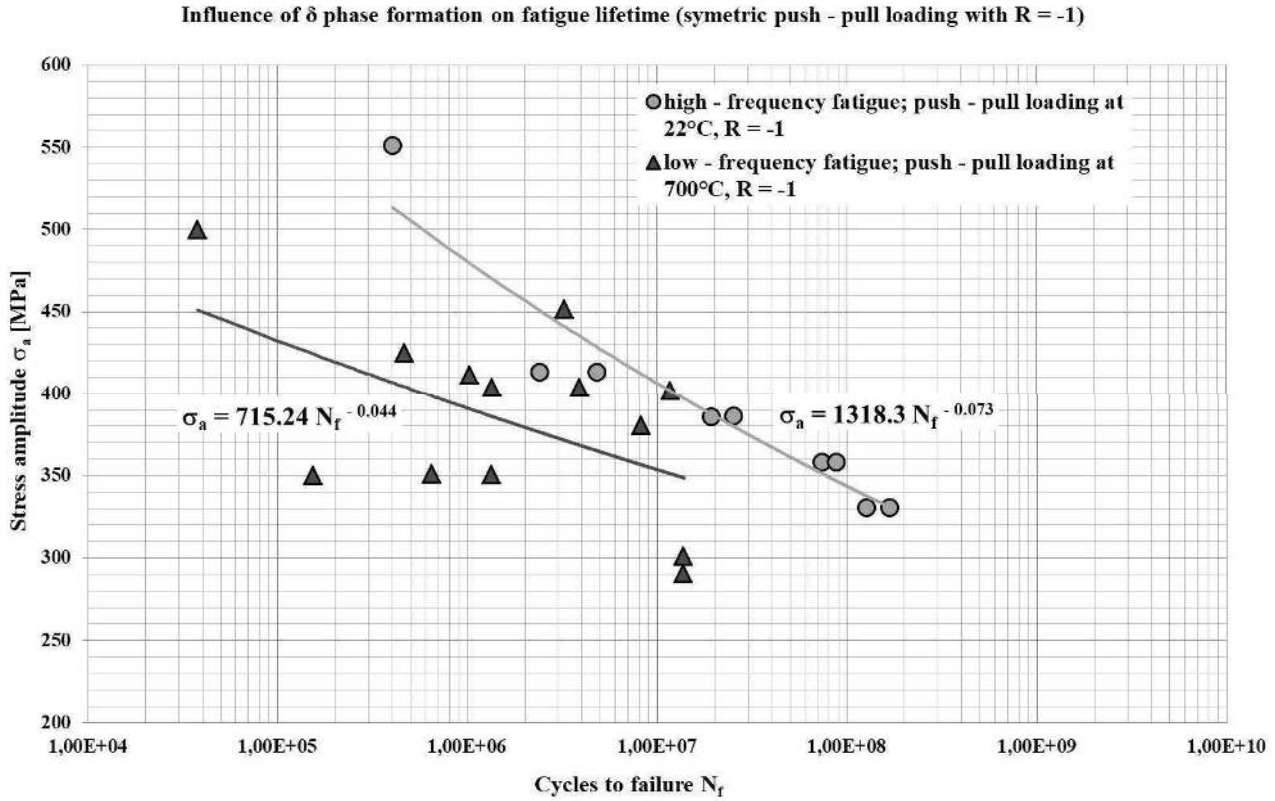

Fig. 8 *The S – N curves for IN 718 compared with various δ phase fraction*

When metastable γ'' phase changes into stable but incoherent δ phase, this δ phase starts to form such Widmanstätten needles inside of grains and helps to crack propagate through the grains – because of loss of coherency with matrix. This fact is demonstrated on Fig. 9, where orientation of grains with Widmanstätten δ phase close to surface (Fig. 9a) and mixed mechanism of fatigue crack propagation can be seen (Fig. 9b).

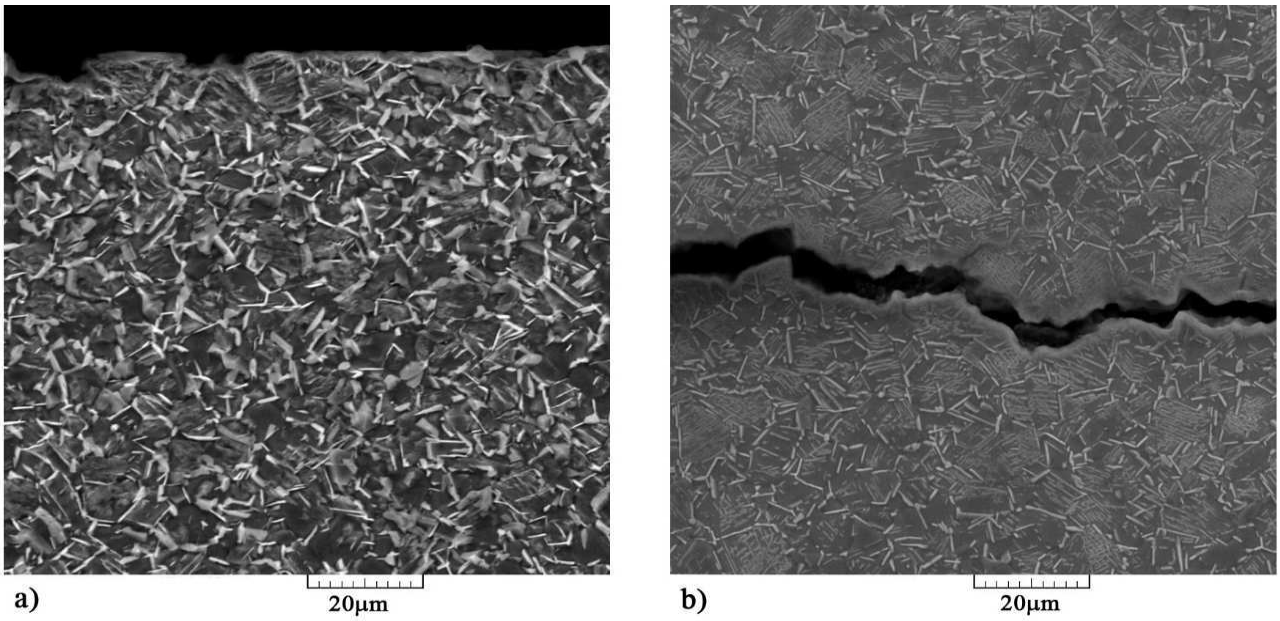

Fig. 9 *SEM micrograph of fatigue crack propagation with Widmanstätten δ phase close to surface (a) and different secondary fatigue crack propagation mechanism (b)*

4 Conclusion

The influence of increased δ phase formation on fatigue lifetime at high temperature in Ni – Cr – Fe IN 718 hardenable alloy was described in this article. From experiments the following conclusions can be drawn:

- the microstructure of IN 718 alloy consists from austenitic γ matrix, MC carbides and fractions of phases such γ'', γ', and about 39.5% of δ phase fractions in starting stage,
- the δ phase was identified with SEM and TEM diffraction and it is situated on grain boundaries or twin boundaries in plate (needle) like shape and blocky shape presented intergranularly respectively,
- applied heat-treatment at 700 °C / 72 hrs. increases the δ phase volume fraction on 52.5% what means conversion of metastable γ'' phase into stable δ phase via mechanism described above - the releasing of Nb into newly formed volumes of δ phase; also coarsening of γ' phase was observed as a sign of coherency loss with γ matrix,
- specimens subjected to push – pull fatigue test at temperature 700 °C (LCLF) show another increasing of δ phase volume fraction to 65%; δ phase transforms into Widmanstätten pattern, what influences negatively on fatigue crack propagation – causes intercrystalline cleavage,
- about the inluence of δ phase formation on fatigue lifetime is from obtained S –N curves clearly seen that increasing δ phase volume from 39.5% in starting stage onto 65% obtained after high temperature fatigue test decreases the fatigue lifetime about 40% (from σ_a = 386 MPa at room temperature to σ_a = 275 MPa at 700°C).

Generally, the influence of δ phase on mechanical properties when situated at grain boundaries in blocky or plate like form in reasonable amount up to 39.5% is positive, δ phase stabilizes the grain size and prevents the grain boundary slip at creep loading or tensile stress even hardness. However, when volume of δ phase increases to 65%, the Widmanstätten pattern is created and the fatigue lifetime is significantly lower. That is the negative influence of higher volume of δ phase on high temperature fatigue properties.

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