Fractography analysis of Inconel 718 fatigued at 700°C

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Abstract. This work deals with the fractography analysis of nickel-base superalloy Inconel 718 fatigued at 700°C in air atmosphere in the high cycle region. During cyclic loading of this alloy at high temperatures some different mechanisms compared to cyclic loading at ambient temperature take place. Cyclic plastic deformation at high temperatures causes some structural changes, which could have some influence on the fatigue process.

Key words – Inconel 718, high temperature fatigue, fractography analysis

1. Introduction

Inconel 718 is precipitation hardenable nickel-base superalloy characterized by excellent mechanical properties in wide range of working temperatures (from -250°C up to 700°C), good corrosion resistance and good weldability. These properties are a result of specific microstructure consisting of gamma matrix (substitution solid solution of alloying elements in nickel), strong coherent precipitates gamma double prime (Ni₃Nb) and gamma prime (Ni₃Al,Ti), delta phase (non-coherent, stable form of Ni₃Nb with orthorhombic lattice) which is located along grain boundaries. In structure there are also carbide and carbonitride particles, which even their considerably small content, and have significant influence on the fatigue process, especially in the high temperature loading regime. The characteristics of the individual structural components are well described in the works of BELAN J. 2015, KALLURI S. ET AL. 1994, DARECKÝ J. 2001, and many others. Cyclic plastic deformation in synergy with high testing temperature can cause a phase transformation in the structure of Inconel 718, which might have significant influence on the fatigue process and its stages. Field of the high cycle fatigue of In 718 at ambient temperature is well described by many authors (BELAN J. ET AL. 2016, DONACHIE M. J. 2002). In this type of loading usually crystallographic initiation takes place in the suitable oriented grain at the surface region. Fatigue crack propagation is transcrystalline through facets of individual grains. High cycle fatigue at high temperature of this alloy is usually investigated up to 650°C, even the highest possible operating temperature for In 718 is 700°C according to manufacturers and published TTT diagrams (RADAVICH J. F. 1989) etc. Therefore, the fatigue
process of Inconel 718 loaded at 700°C is not well explored. Another important factor is that most published papers about high temperature fatigue of In 718 were carried out by considerably low frequency testing machines where the effect of environment (oxidation at high temperatures) has strong impact on the fatigue test. According to the study of (ABIKHI M. ET AL. 2013), during high temperature cyclic loading significant changes in the initiation mechanisms can occur. During the high temperature cyclic loading at small frequencies initiation on the carbidic and carbonitridic particles was observed, which is caused by a phenomenon called oxidation assisted crack initiation which is well described in the study of (ABIKHI M. ET AL. 2013). In very special cases, when the oxidation attack on the grain boundaries is large enough, the change of the fatigue crack propagation can occur, and the intercrystalline crack propagation could be seen in the fracture surfaces. The aim of this study is to describe fatigue process of alloy Inconel 718 in terms fractography of the fracture surfaces after cyclic loading at 700°C, at an average frequency of 87Hz, which is according to published papers, high enough to eliminate the influence of the environment.

2. Experiment

To perform fatigue process nickel-based alloy Inconel 718 in form of rods was used, from which specimens were machined. Material was heat treated solution annealed at 980°C/1h and aged at 720°C/8h and subsequent cooled to 620°C and hold 8h at this temperature. Chemical composition and the mechanical properties are shown in the Table 1 and Table 2.

Table 1. Chemical composition of tested material

|        | In 718 | Ni | Cr  | Fe  | Nb | Mo | Ti | Al | Co |
|--------|--------|----|-----|-----|----|----|----|----|----|
| wt%    |        | 52.2 | 18.5 | 5.3 | 3.04 | 1 | 0.52 | 0.13 |
| wt%    |        | 0.03 | 0.04 | 0.06 | 0.008 | <0.001 | 0.004 | 0.03 |

Table 2. Mechanical properties of In 718

| Temperature | Ultimate tensile strength [MPa] | Yield strength [MPa] | Elongation [%] | Hardness HBW |
|-------------|---------------------------------|---------------------|----------------|--------------|
| 20°C        | 1238                            | 1474                | 26             | 450          |
| 650°C       | 1030                            | 1211                | 13.5           | -            |

Fatigue test was performed with using electromagnetic pulsator, ZWICK ROELL AMSLER HFP 5100. Specimens were cyclically loaded by symmetric tension-compression loading (R=1), at 700°C, in air atmosphere. Fatigue test has been conducted at stress-controlled regime.

From the broken specimens a specimen for microscopy observation was manufactured. The aim of this observation was to compare the microstructure of the initial state with the microstructure after cyclic loading at 700°C. Specimens for the light and electron microscopy observations were prepared by standard process for metallographic samples preparation and were electrolytic etched in the solution of 10g CrO3 in 100ml H2O.

Fracture surfaces of the specimens broken by the cyclic loading at 700°C were evaluated by SEM, with the aim of describing mechanisms of stages of the fatigue process (crack initiation, crack propagation and final rupture). Fracture profiles were also evaluated by the light microscopy, the dependence of the roughness on the length of the crack (and thus on the \( K_I \)) was evaluated.

3. Results and discussions

The high temperature, at which fatigue test was performed caused significant changes in the fatigue process. At fracture surfaces of the broken specimens was clearly visible changes in the fatigue crack initiation and propagation. During cyclic loading of In 718 at 700°C, multiply initiation of fatigue crack was observed. While the number of initiation sites depends on the value of loading stress, when at the specimens loaded at higher stress level higher number of initiation sites was observed. On the fracture surface a great number of secondary cracks was present, while the value of these cracks depends on the loading stress, same as the number of initiation sites. On the fracture surfaces there was no visible macroscopic clear border between the fatigue crack propagation area and the area of the final fracture.

Initiation of the fatigue crack takes place solely on the carbidic or carbonitridic particles at surfaces or subsurface area (Fig. 1 and 2), which is in clear contrast with the crystallographic initiation observed at fracture surfaces of specimens fatigued at ambient temperatures. In the study of (ABIKHI M. ET AL. 2013)
was well described mechanism of crack initiation of carbide particles at specimens cyclically loaded at high temperature at small frequencies of loading (oxidation and subsequent volume expansion of carbide particles caused internal stresses in the matrix, which serves as a preferred initiation places). Implementation of this mechanism in the study is questionable, and it needs some more research to confirm or reject this claim, but in general, for example, the specimen broken after $N_f = 3.6 \times 10^6$ cycles, had quite sufficient time to oxidation of carbide particles, because in the high cycle fatigue, initiation stage consume more than 90% of the whole time of the fatigue test and duration of test was 7.5 hours.

Fig. 1. Typical initiation site, In 718 cyclically loaded at 700°C, REM.

Fig. 2. Detail of the initiation place, there is clearly visible that initiation of fatigue crack took place on the carbonitridic particle, REM.

Fig. 3. Area of fatigue crack propagation, there is visible rough character of fracture surface, REM.
Fatigue crack propagation occurs by the transcry stalline ductile mechanism (Fig. 3), while on the fracture surface a great number of the secondary cracks was observed. Facets of intercrystalline fatigue crack propagation weren’t observed in neither case. Formation of intercrystalline fracture is in the case of In 718 is conditional on the present the high content of laves phase (which is not possible in the wrought material) or by very high level of oxidation of the grain boundaries region, but this case is possible only when specimens are loaded at very low frequencies. On the fracture surfaces, in the crack propagation area striations could be observed, but they were hard to detect due to a certain degree of oxidation at the surface and a significant roughness topography of the fracture surfaces. Fracture profiles observations showed, that by increasing length of the crack (and thus $K_\sigma$), the fracture profiles had higher roughness, which is well documented on Fig. 5 and 6.

Final rupture took places by the transcrystalline ductile mechanism with very strong dimple morphology (Fig. 4), which was caused increased ductility in testing temperature. Facets of intercrystalline fracture were not observed in the final rupture region, and it shows a good state of microstructure, even some structural changes caused by cyclic plastic deformation at 700°C, which are described in the next part.

Microstructural observation showed that the cyclic plastic deformation at high temperature (700°C) caused significant microstructural changes. Microstructure of the initial state (Fig. 7) consists of the fine grains of gamma matrix, delta phase particles at grain boundaries and with a primary carbides Nb(Ti)C and carbonitridic particles TiNC. In structure there are also fine precipitates gamma prime and gamma double prime, but these are not detectable by this kind of observation due their small dimensions and the strong coherency with the matrix. In the microstructure of the specimen cyclically loaded at 700°C (Fig. 8) were observed significant changes, especially in the form and the amount of the delta phase particles. Cyclic plastic deformation caused mechanical dissolving of gamma double prime particles (this mechanism is described in the work (WORTHEM D.W. ET AL. 1990), which leads to faster transformation of gamma double
prime to the intragranular delta phase (delta precipitate as a plates – in the cross section needles). Despite the fact that the volume of delta phase in the structure dramatically increased, it could be stated that it has no effect of the fatigue process in the Inconel 718 superalloy.

4. Conclusions

Based on the carried out fatigue test and subsequent observation of Inconel 718 cyclically loaded at 700°C following conclusions can be drawn:

- Initiation of the fatigue crack in Inconel 718 fatigued at 700°C occurs on the carbide/carbonitridic particles at the surface or near the surface.
- On the specimens was observed to multiply initiation, when the higher loading stress result in more initiation sites.
- Fatigue crack propagation occurs by the transcryrstalline ductile mechanism, while on the fracture surfaces no facets of intercrystalline crack propagation were recorded.
- Fracture surface contains a large amount of secondary crack, when the higher loading stress results in higher number of such crack.
- There were recorded significant changes in the microstructure caused by cyclic plastic deformation at high temperature (increase of the amount of the delta particles), but no effect of this changes on the fatigue process was observed.

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Literature

1. BELAN J. 2015. High Frequency Fatigue Test of In 718 Alloy – Microstructure and Fractography Evaluation. In: Metalurgija 54 (2015)1, p. 59-62. Zagreb.
2. BELAN J. ET AL. 2016. Fatigue Test of the Inconel Alloy 718 Under Three Point Bending Load at Low Frequency. In: Advanced Structured Materials. Vol. 33, p. 75-84.
3. KALLURI S. ET AL. 1994. Deformation and Damage Mechanisms in Inconel 718 Superalloy. In: Superalloys 718,625 and various derivates. Ed. E. A. Loria. The Minerals. Metals & Materials society, 1994, p. 593-606.
4. DARECKÝ J. 2001. Superzliatiny niklu a ich obrábanie. EDIS-ŽU in Žilina (in Slovak).
5. DONACHIE M. J. DONACHIE S. J. 2002. Superalloys – A technical Guide. ASM International, USA.
6. RADAVICH J. F. 1989. The Physical Metallurgy of Cast and Wrought alloy 718. In: Superalloy 718 – Metallurgy and Applications, Ed.: E. A. Loria, The Minerals, Metals & Materials Society 1989, p. 229-240.
7. ABIKCHI M. ET AL. 2013. Fatigue Life and Initiation Mechanisms in Wrought Inconel 718 DA for Different Microstructures. 13th International Conference on Fracture, 2013. p. 1-11.
8. WORTHEM D. W. ET AL. 1990. Inhomogenous Deformation in Inconel 718 During Monotonic and Cyclic Loadings. In: Metallurgical and Materials Transactions 21A, 1990, p. 3215-3220.
9. COZAR R. ET AL. 1989. Effect of Environment and Microstructure on the High Temperature Behavior of Alloy 718. In: Superalloys 718 – Metallurgy and Applications. Ed.: E. A. Loria. The Minerals, Metals & Materials Society 1989, p. 241-256.