# PREDICTION OF MAXIMUM CASTING DEFECT SIZE IN MAR-M 247 ALLOY PROCESSED BY HOT ISOSTATIC PRESSING

Miroslav Šmíd[1,*], Stanislava Fintová[1,2], Ludvík Kunz[1], Pavel Hutař[1], Karel Hrbáček[3]

[1] Institute of Physics of Materials Academy of Sciences of the Czech Republic v. v. i., Žižkova 22, 616 62 Brno, Czech Republic
[2] Brno University of Technology, CEITEC BUT – Central European Institute of Technology, Technická 3058/10, 616 00 Brno, Czech Republic
[3] První brněnská strojírna Velká Bíteš a.s., Vlkovská 279, 595 12 Velká Bíteš, Czech Republic

[*]corresponding author: tel.: +420 532 290 362, fax: +420 541218 657, e-mail: smid@ipm.cz

***Resume***

Nickel based MAR-M 247 superalloy treated by hot isostatic pressing was investigated with the aim to identify the influence of casting defect size on fatigue life. Two testing temperatures of 650 and 800 °C and one stress amplitude were chosen for fatigue tests. The Murakami approach and the largest extreme value distribution theory were applied. It has been found that the maximum size of casting defects in a specimen can be satisfactorily predicted. Fatigue life of specimens was in good agreement with assumptions based on the evaluation and prediction of the casting defect size.

Available online: http://fstroj.uniza.sk/journal-mi/PDF/2015/04-2015.pdf

***Article info***

*Article history:*
Received 25 August 2014
Accepted 4 February 2015
Online 17 February 2015

*Keywords:*
MAR-M 247;
Superalloys;
Fatigue;
Casting defects;
Elevated temperatures.

ISSN 1335-0803 (print version)
ISSN 1338-6174 (online version)

## 1. Introduction

Nickel based superalloys has been one of the most essential class of materials in aerospace, power generation and automotive industries since many decades. These alloys are irreplaceable in many applications despite lot of new materials were developed [1]. Their combination of mechanical properties and corrosion and oxidation resistance is still unique. That is why superalloys are commonly used for the most critical parts of turbines [2 – 4].

MAR-M 247 is a well-known advanced superalloy suitable for investment casting. Mechanical properties are mainly determined by the structure of the material. Superalloys typically consist of a $\gamma$ matrix, coherently embedded strengthening precipitates of a $\gamma'$ ordered intermetallic phase and carbides. The MAR-M 247 alloy contains about 60 % $\gamma'$ phase volume fraction which provides very good high temperature mechanical properties [5]. Solid solution alloying of the $\gamma$ matrix by W, Ta, Mo and other elements enhances strength and thermal stability while carbides are beneficial for grain boundary strength. A special feature of this alloy is the addition of Hf which has an enhancing effect on tensile strength and temperature retention of $\gamma'$ [6]. While processed under optimal casting conditions and with well-chosen heat treatment, the alloy exhibits high tensile strength and creep resistance at high temperatures along with excellent oxidation resistance. Thanks to the mentioned combination of properties the MAR-M 247 alloy is used in turbine components subjected to various loading and high temperatures [7 – 9].

Casting defects like shrinkage pores and gas pores are an inevitable feature of every cast material. Their size and distribution is strongly dependent on casting conditions. These irregularities act as stress concentrators and fatigue crack initiation sites. Hot Isostatic Pressing (HIP), which can diminish pores and casting defects, improves the mechanical properties [10]. However, this process can be accompanied by coarsening of the $\gamma'$ phase which can cause a decrease of the yield strength [11] or in some cases coarsening of dangerous carbide particles [12]. Despite the shrinkage pore size is usually in the range of tenths or hundreds of micrometers they are difficult to detect by non-destructive defectoscopy. Therefore these material defects can cause significant scatter in service life-time [13] and also unexpected failures of turbine components. This serious material issue has been studied for decades and it is still of great interest for the engineering and scientific community. Therefore investigation focused on relationship of the fatigue life and the porosity of material is very desirable.

Statistical analysis of casting defects present in the structure has been shown [14] to be an useful tool in the procedure of the fatigue life prediction. This method was used for fatigue life prediction of different materials and in some cases a quite good agreement with fatigue test results [13, 15 – 17] was demonstrated.

The present paper is focused on the statistical analysis of the casting defects of Ni-based superalloy MAR-M 247 after HIP. The analysis of casting defects was performed according to Murakami's statistical method using Largest Extreme Distribution theory (LEVD). This theory enables to predict the largest defects which can occur in a given volume. The predicted largest defect sizes were compared to the sizes of the defects observed on the fracture surface of the failed specimens. Proposed fatigue life according to the statistical evaluation and prediction of casting defects was found to be in agreement with the number of cycles reached by fatigue tests conducted at 650 °C and 800 °C.

## 2. Experimental material and methods

Experimental material was provided by PBS Velká Bíteš, a.s. as pre-casted specimens. Final machining of the cylindrical specimens was done to obtain the gauge length diameter of 8 mm. The chemical composition of MAR-M 247 guaranteed by the producer is shown in the Table 1.

Pre-casted specimens were subjected to HIP treatment at 1 200 °C at 100 MPa for 4 hours before final machining in order to reduce the extent of casting defects. Subsequently, the alloy was heat treated by homogenizing annealing at 1 200 °C for 2 hours and following precipitation annealing at 870 °C for 24 hours.

Fatigue tests were performed under controlled load in symmetrical tension-compression loading (load ratio $R$ = -1). A resonant fatigue machine Roell Amsler equipped with VibroWin electronics was used for fatigue tests. The full load amplitude was reached by a ramp during several hundreds of loading cycles. The test frequency was 120 ± 3 Hz. Fatigue tests were held until final failure of the specimen or decrease of test frequency higher than 5 Hz. The heating to the desired experimental temperature was provided by an electric furnace with temperature stability on gauge length within ± 1 °C.

*Table 1*

*Chemical composition of MAR-M 247 alloy (wt. %).*

| C | Cr | Mo | Al | Ti | Ta | W | Co | O | N | Nb | B | Hf | Ni |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.15 | 8.37 | 0.67 | 5.42 | 1.01 | 3.05 | 9.92 | 9.91 | 5 ppm | 6 ppm | 0.04 | 0.015 | 1.37 | rest |

The fatigue tests in this study were performed at two temperatures, 650 °C and 800 °C. For identification of the influence of casting defects and temperature all tests were performed at one stress amplitude, namely at 240 MPa.

Metallographic specimens for microstructural observations and the statistical analysis of casting defects were machined from fatigued specimens by electroerosion cutting. The cutting of the gauge length of all specimens was parallel to the loading direction. Subsequently, the surface of the sections was carefully prepared by mechanical grinding with grind papers and polishing with diamond paste. The last finalizing step was done by vibration polishing. Observation of the metallographic samples and fracture surfaces of fatigued specimens was performed using Scanning Electron Microscope (SEM) Tescan LYRA 3 XMU. The size of the casting defects was determined by light microscopy (LM) using confocal microscope Olympus LEXT OLS3100.

For the analysis of defects the LEVD theory proposed by Murakami [14] was used. The theory is based on the evaluation of the size of the largest defects on the controlled area $S_0$ (field of view). The defect size was characterized in terms of the square root of its area, $area^{1/2}$. The measurement was done on $n$ number of the controlled areas. The number of the controlled areas has to be sufficient for reasonable statistical description. On each metallographic specimen at least 30 measurements were done. The number of the measurements is dependent on specimen size and on chosen magnification. For this paper a magnification of 480× was used. According to the return period $T = S/S_0$ which is based on measured sizes of the largest defects found on the controlled area, it is possible to predict defect sizes on areas larger than the controlled area.

## 3. Results

### *3.1 Microstructural analysis*

Metallographic specimens were prepared from the specimens used for the high temperature fatigue testing. Typical microstructure of the examined MAR-M 247 superalloy is shown in Fig. 1. The alloy exhibits coarse dendritic structure from the macroscopic point of view.

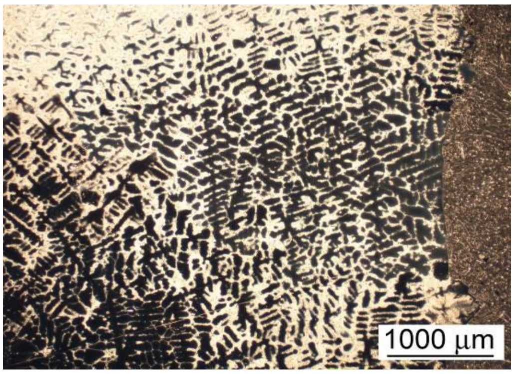



*Fig. 1. Typical microstructure of MAR-M 247, LM, etch. 40 ml $HNO_3$ + 30 ml HF, dark field.*
*(full colour version available online)*

The microstructure of the alloy is significantly heterogeneous in terms of size and morphology of $\gamma'$ strengthening precipitates (see Fig. 2).

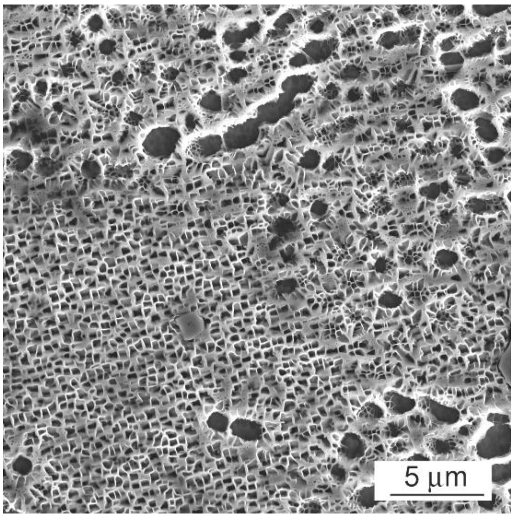



*Fig. 2. Size and shape of the strengthening $\gamma'$ precipitates, SEM, etch. 40 ml $HNO_3$ + 30 ml HF.*

Areas of fine cubic $\gamma'$ and coarse $\gamma'$ phase with irregular shape were found to be characteristic for this material. Average edge size of the cubic precipitates was 0.4 μm and the average diameter of coarse precipitates was 1.6 μm.

The structure contains numerous casting defects. The characteristic size and morphology of the defects is documented in Fig. 3. The defects have mostly spherical shape; however, some irregularly shaped shrinkage pores were present in the structure even after HIP.

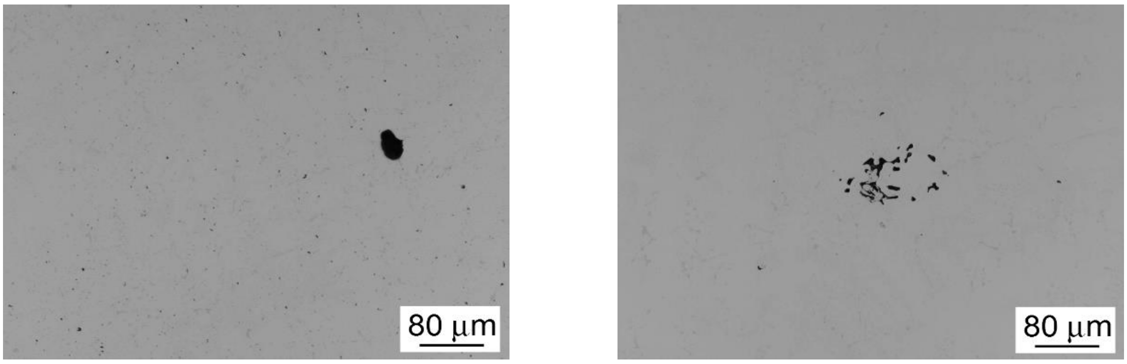
### *3.2 Fatigue test results*

All specimens were tested at stress amplitude $\sigma_a$ = 240 MPa. Results of the performed fatigue tests are shown in Table 2. The specimens were arranged in the table according to the number of cycles till fracture. Range of reached number of cycles to fracture in case of the specimens tested at 650 °C was from 7.83×10$^5$ to 1.3×10$^6$ cycles. Fatigue tests conducted at 800 °C revealed an increase in scatter of fatigue life data while the range of number of cycles to fracture was 4.65×10$^5$ to 8.3×10$^6$ cycles. It should be noted that the average fatigue life increased with higher experimental temperature.

Fractographic observation revealed the presence of casting defects on all fracture surfaces.

### *3.3 Analysis of casting defect distribution*

The evaluation of defect size and distribution was performed on metallographic sections prepared from the cross-section of the gauge length of specimens after fatigue testing. The predictions of the largest defect size which can occur on an area *S*, which is equivalent to the area of the cross section of gauge length of the fatigued specimen ($S$ = 50 mm$^2$), were carried out. The results were compared with defects responsible for the fatigue crack initiation in particular specimens. The size of these defects was evaluated on fracture surfaces of failed specimens.




*a) rounded defects* *b) shrinkages*

*Fig. 3. Typical casting defects present in the microstructure, LM.*

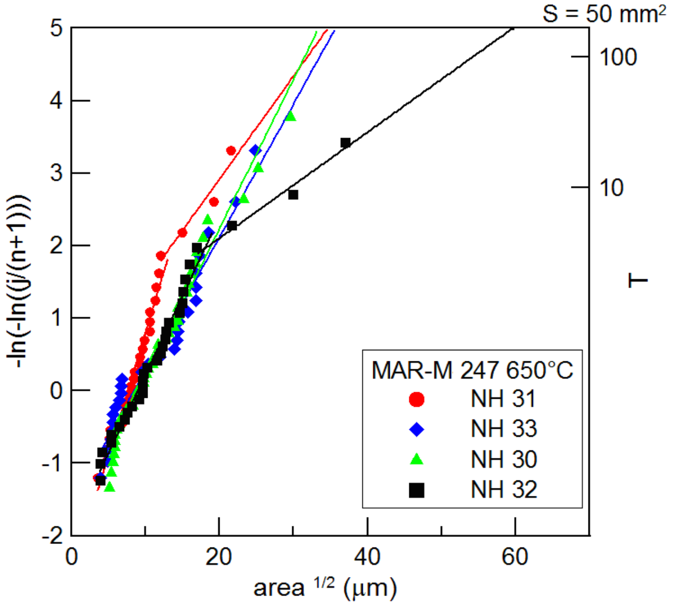
*Table 2*

*Fatigue tests results.*

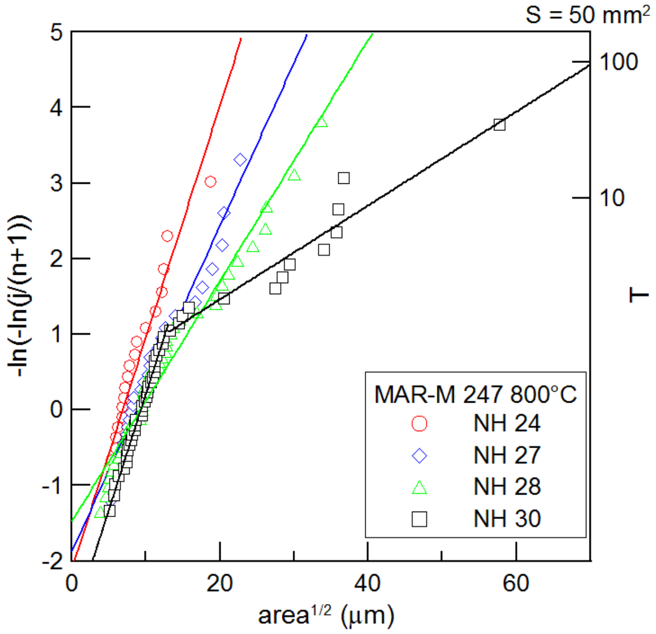
| **650 °C specimen** | **$\sigma_a$ = 240 MPa $N_f$ (cyles)** | **800 °C specimen** | **$\sigma_a$ = 240 MPa $N_f$ (cycles)** |
|---|---|---|---|
| NH 31 | 1 301 000 | NH 24 | 8 309 000 |
| NH 33 | 1 132 000 | NH 27 | 1 991 000 |
| NH 30 | 987 000 | NH 28 | 1 078 000 |
| NH 32 | 783 000 | NH 34 | 465 000 |

The results of analysis using the LEVD theory are shown in Fig. 4. The size of the largest defects measured on evaluated specimens is presented as a Gumbel plot. The data were extrapolated for the values corresponding to the largest defects which could occur in area of $S$ = 50 mm$^2$ marked on the T axis. The ratio between the projected area and the controlled area is $T = S/S_0 = 163$ in this study.

The results of the prediction of the largest size of defects which can occur on the cross section of the gauge length and experimentally determined areas of defects observed on the fracture surface of particular specimens are shown in Table 3. The size is presented in terms of area of the defect.




*a) specimens tested at 650 °C*




*b) specimens tested at 800 °C*

*Fig. 4. LEVD plots of porosity.*

*(full colour version available online)*

*Table 3*
*Values of the average defect size, the predicted largest defect size and real defect size found on fracture surface of fatigued specimens.*

| 650 °C | | | | 800 °C | | | |
|---|---|---|---|---|---|---|---|
| **specimen** | **average defect size ($\mu m^2$)** | **predicted largest defect size ($\mu m^2$)** | **real defect size on the fracture surface ($\mu m^2$)** | **specimen** | **average defect size ($\mu m^2$)** | **predicted largest defect size ($\mu m^2$)** | **real defect size on the fracture surface ($\mu m^2$)** |
| **NH 31** | 107 | 1 100 | 1 200 | **NH 24** | 84 | 550 | 560 |
| **NH 33** | 162 | 1 142 | 740 | **NH 27** | 149 | 1 044 | 3 940 |
| **NH 30** | 169 | 1 145 | 490 | **NH 28** | 211 | 1 700 | 1 160 |
| **NH 32** | 199 | 3 884 | 40 | **NH 34** | 342 | 5 608 | 4 870 |

In Table 3 are further given the values of the average size of the largest defects measured directly on the metallographic specimens.

## 4. Discussion

Cast materials, like the MAR-M 247 examined in this study, contain inevitable casting defects in some extent. The size of the defects can be minimized by the HIP process. However, it is not possible to completely eliminate some shrinkage pores which are too large. Even after HIP they can be larger than the size of intermetallic phases and detrimental for fatigue strength of the material.

As it was mentioned above, Murakami's statistical LEVD theory (for more details see [14]) was used in this study. The porosity of fatigued specimens was evaluated and the largest size of defects which could occur in the area larger than the examined area was predicted. The average defect size for examined metallographical specimens was in the range from 84 to 342 $\mu m^2$ (see Table 3). Measured defect sizes are smaller than the size of defects observed by Roskosz et al. [19] who examined the porosity in precision cast blades of aircraft engine from the MAR-M 247 alloy. The size of defects range observed in their study was from 135 to 2745 $\mu m^2$. The difference in the average defect size can be explained by the applied HIP process. While Roskosz et al. in [19] observed mostly defects of shrinkage shape in this study while the majority of them were predominantly rounded.

The porosity evaluation was conducted on metallographical specimens taken from each fatigued specimen. Characteristic values of the defect size were subsequently compared with the fatigue test results obtained at 650 °C and 800 °C. The number of cycles reached by the fatigued specimens is in good agreement with the porosity of the individual metallographic specimens and with the predicted largest defect sizes for the area of the cross section of the specimens (see Table 2 and 3). Also the scatter of the obtained number of cycles to fracture at the same stress amplitude at chosen temperatures correlates well with the different porosity of individual specimens. It should be noted that specimens were prepared by the same technology and are different only in the size and density of defects. Just these differences were the reason for the various fatigue life of individual specimens. Fig. 5 summarizes obtained relationship of the predicted largest defect sizes of the area with obtained fatigue life-times. Especially at 800 °C very good agreement in these 2 parameters is clearly visible. The correlation of the results is worse at temperature 650 °C which is caused by low number of examined specimens and also by different fatigue crack propagation mechanisms observed at this temperature.

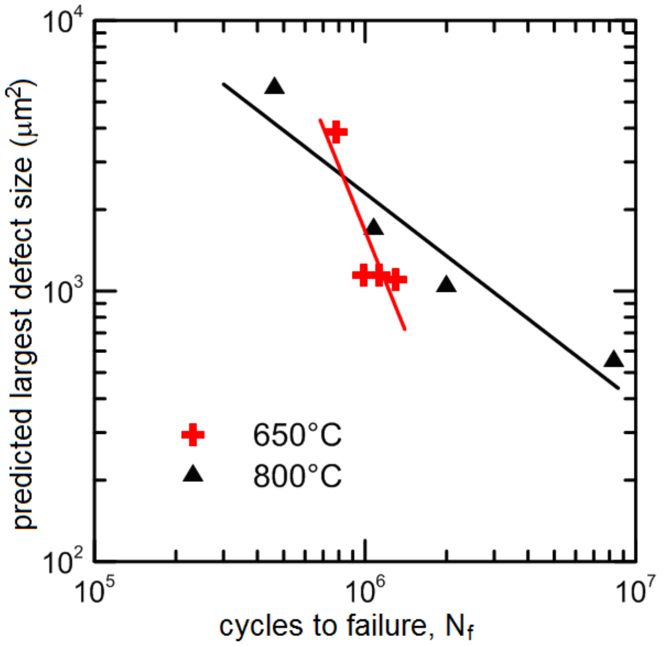



*Fig. 5. Relation between predicted largest defect size and obtained fatigue life-time for given specimen.*
*(full colour version available online)*

Despite these facts, good agreement would be reached even on this temperature with reasonably higher number of observations.

The prediction and the largest defect size was not in agreement in the case of the specimens NH31 tested at 650 °C and NH27 tested at 800 °C. The reason for this discrepancy can be in difficult distinguishing of the real size of shrinkage pore. However, also in these cases the number of cycles to fracture is in reasonable correlation with the predicted largest defect sizes for cross section area of the fatigue specimen.

Metallographic sections on which the analysis of defects was performed are showing just 2D information about structure and porosity. On the other hand, especially the shrinkage pores are complicated 3D objects and therefore a metallographic specimen describing one cross section is not sufficient to fully represent the observed 3D structure. Therefore, for more accurate results it would be necessary to investigate a sufficient number of metallographical specimens with different orientation. The influence of cutting of the shrinkages when the metallographic specimen is prepared was studied in [16]. Nevertheless, the presented paper shows that also the used simplified procedure brings reasonable and useful results.

The performed study confirms that the approach by Murakami, [14], based just on the metallographic analysis and the prediction of the largest defect size seems to be a suitable treatment for prediction of fatigue life of the MAR-M 247 superalloy.

## 5. Conclusion

Maximum size of casting defects in a specimen from the MAR-M 247 alloy was satisfactorily predicted on the basis of an analysis of defect distribution on small metallographic sections detracted from the specimen gage length. It has been shown that the fatigue lives of tested specimens are in good agreement with the predicted largest defect size. The specimens with the largest predicted defect size had the shortest fatigue lives and vice versa.

***Acknowledgements***

*This research was financially supported by the project FR-TI4/030 of the Ministry* *of Industry and Trade of the Czech Republic and by the grants No. CZ.1.07/2.3.00/20.0214 and CZ.1.07/2.3.00/30.0039 of the Ministry of Education, Youth and Sports of the Czech Republic.*

**References**

[1] M. Ashby: Materials Selection in Mechanical Design, 3rd edition, Butterworth-Heinemann, Oxford 2005.

[2] M.J. Donachie, S.J. Donachie: Superalloys – A Technical Guide, 2nd edition, ASM International, USA 2002.

[3] R.C. Reed: The Superalloys, Fundamentals and Applications, 2nd edition, Cambridge University Press, New York 2008.

[4] A. Pineau, S.D. Antalovich: Eng. Fail. Anal. 16(8) (2009) 2668-2697.

[5] C.M. Sonsino, U. Brandt, J. Bergmann: In: 3rd International Conference on Low Cycle Fatigue and Elasto-Plastic Behaviour of Materials, Ed.: K.T. Rie et al., Berlin 1992, pp. 262-268.

[6] N. Baluc, R. Schäublin.: Philos. Mag. A. 20(1) (1996) 113-136.

[7] C.A. MacIntyre, P. N. Agarwal: In: Aerospace Congress and Exposition, Warrendale, Long Beach, California 1984, paper 841515, pp. 35-45.

[8] I.R. Delgado, G.R. Halforf, B.M. Steinetz, C.M. Rimnac: In: Proceedings of the ASME Turbo Expo Vol. 5, Montreal, Canada 2007, pp. 583-597.

[9] M. Kaufman: In: Superalloys 1984, Ed.: M. Gel et al., Metallurgical Society of AIME, Warrendale, 1984, pp. 43-52.

[10] H.V. Atkinson, S. Davies: Metall. Mater. Trans. A 31A(12) (2000) 2981-3000.

[11] H.Y. Bor, C. Hsub, C.N. Wie: Mater. Chem. Phys. 84 (2004) 284–290.

[12] K.C. Antony, J.F. Radavich: In: Superalloys 1980, 4th Int. Symp. on Superalloys, Ed.: J.K. Tien et al., Metal Park, Ohio 1980, pp. 257-265.

[13] L. Kunz, P. Lukáš, R. Konečná: Int. J. Fatigue 32(6) (2010) 908-913.

[14] Y. Murakami: Metal Fatigue: Effects of Small Defects and Nonmetallic Inclusions, 1st edition, Elsevier, Oxford 2002.

[15] L. Kunz, P. Lukáš, R. Konečná, S. Fintová: Int. J. Fatigue 41 (2012) 47-51.

[16] G. Nicoletto. R. Konečná, S. Fintová: Int. J. Fatigue 41 (2012) 39–46.

[17] M. Filippini, S. Beretta, L. Patriarca, G. Pasquero, S. Sabbadini: J. ASTM Int. 9(5) (2012) 279-295.

[18] L. Kunz, P. Lukáš, R. Konečná: Eng. Fract. Mech. 77 (11) (2010) 2008-2015.

[19] S. Roskosz, M. Staszewski, J. Cwajna: Mat. Chract. 56(4-5) (2006) 405–413.