Rare Met. (2019) 38(7):642–652
https://doi.org/10.1007/s12598-018-1123-x

RARE METALS
www.editorialmanager.com/rmet

CrossMark

Mechanism of high-temperature oxidation effects in fatigue crack propagation and fracture mode for FGH97 superalloy

Chao Xu, Zhi-Hao Yao*, Jian-Xin Dong, Yu-Kun Jiang

Received: 30 August 2017 / Revised: 17 December 2017 / Accepted: 7 July 2018 / Published online: 4 September 2018

Abstract The high-temperature fatigue crack growth behaviors in powder metallurgy (P/M) Ni-based superalloy FGH97 for turbine disk application were investigated at different temperatures (650, 700 and 800 °C) in air using a combination a servohydraulic test system, fractographic and microanalytical investigations. It is found that there is a temperature-sensitive region in which the fatigue life of FGH97 alloy decreases sharply. To further evaluate the crack propagation mode and oxidation effects, interruption experiments were conducted at 700 and 300 °C, respectively. The results indicate that the reduction of the fatigue lifetime for FGH97 takes place when the fracture mechanism transforms from a predominantly transgranular mode to an intergranular one as the temperature increases. Although the microstructures and mechanical properties may vary with the temperature, they are not the dominating factors contributing to the temperature sensitivity of fatigue property for FGH97. It is the oxidation that governs the fatigue crack growth behaviors in air at elevated temperature. The enhanced thermal activity of oxygen and certain active metal elements result in accelerated oxidation reaction. The brittle oxide intrusions formed at the crack tip and grain boundaries of crack frontier lead to grain boundary weakness, which is responsible for the transformation of crack growth mode and degradation of the fatigue property of FGH97 alloy.

Keywords FGH97; Fatigue crack growth; Temperature sensitivity; Fracture mode; Oxidation

C. Xu, Z.-H. Yao*, J.-X. Dong, Y.-K. Jiang
School of Materials Science and Engineering, University of Science and Technology Beijing, Beijing 100083, China
e-mail: xc158158@163.com

1 Introduction

Powder metallurgy (P/M) Ni-based superalloys have been often used for many high-temperature components in land-based gas turbine, aircraft engine industries, and chemical process industries [1, 2]. The wide use of these materials is based on an exceptional combination of the high-temperature strength, outstanding creep resistance and corrosion resistance, low crack growth rate and stability in long period of service at elevated temperatures. With the development of aviation industry, a higher thrust-weight ratio and a longer fatigue life are desired; hence, the P/M Ni-based superalloys ought to have better mechanical properties and excellent damage tolerance capability.

The key components of aero-engine operating at a harsh environment have to endure high temperature, high-pressure and serious corrosion atmosphere [3–5]. Fatigue failure is the most common reason that causes accidents. It has been estimated that over 80% material failure accidents are caused by fatigue [6], among which the thermal fatigue failures accounts for a large proportion.

Among various factors that affect thermal fatigue resistance of Ni-based superalloys, temperature is a significant one; it influences the fatigue property in many ways [7, 8]. For example, the mechanical properties and microstructures vary with the temperature, elevating temperature weakens the strength of superalloys and promotes crack initiation and growth [7, 9]. In addition, oxidation damage is a concern for turbine disk exposed to engine gas environment for prolonged periods. At elevated temperature, the surface and grain boundaries of superalloys will be oxidized easily [10–12], brittle oxides formed at the grain boundaries lead to a decrease in mechanical strength and an

Table 1 Nominal chemical compositions of FGH97 (Ni being matrix)

C	Cr	Mo	W	Al	Ti	Co	Nb	Zr	B	Hf
0.045	9.020	3.760	4.960	4.910	1.740	15.690	2.590	0.017	0.012	0.300

increasing tendency to intergranular fracture, thereby reducing the fatigue life [11, 13–15].

Zheng [16] found that the fatigue crack scarcely grows within a lower temperature range between 200 and 350 °C. Once the temperature exceeded a critical value, crack would grow in a high speed and fatigue life declined instantly. A similar rule was also found by investigating the effect of temperature on the fatigue crack propagation behaviors for FGH97 alloy. The fatigue crack growth rate (FCGR) increased with temperature increasing from 650 to 800 °C [17]; nevertheless, the increase in amplitude in FCGR between two temperatures obviously depended on the temperature variation range. The difference of FCGR from 750 to 800 °C was significantly larger than that between the two other lower adjacent temperatures. Zhong et al. [18] studied the influence of temperature on FCGR for a novel superalloy TMW-2 and found that the crack growth rate increased sharply with temperature. The above studies indicate that there seems to have a sensitive region of temperature, once the superalloy components service in this region, fracture failure will take place promptly, which poses a huge threat to aircraft and equipment and may result in significant economic loss.

For now, there are a lot of researches at home and abroad, involving the impact of temperature on the fatigue crack propagation behavior of superalloys; however, the research with respect to temperature sensitivity and its fundamental principles are not systematic. Thus, the fatigue crack growth behaviors of FGH97 at different temperatures were analyzed firstly in this paper. The mechanical properties, microstructures and fatigue surface morphologies of FGH97 specimens tested at different temperatures were observed. The interruption experiments were carried out to investigate the crack propagation mode and oxidation at the crack tips and grain boundaries of the samples. Finally, the mechanism of the existence of temperature-sensitive region and the transition law of fatigue fractures were concluded.

2 Experimental

2.1 Materials

The material used in the investigation was P/M nickel-based superalloy FGH97 developed in China for turbine disk application, and similar to Russian EP741NP superalloy except for the optimized composition, Table 1 gives the nominal chemical composition of the alloy. FGH97 alloy was manufactured through plasma rotating electrode process (PREP) milling + hot isostatic pressing (HIP) method [19], and followed by the heat treatment of 1200 °C × 8 h/FC + 1170 °C/AC + 870 °C × 32 h/AC, where FC and AC refer to furnace cooling and air cooling, respectively.

The FGH97 samples underwent traditional metallographic procedure using silicon-carbon paper for grinding from 60 up to 2000 grits, followed by polishing with 2.5- and 0.5-μm diamond paste. The polished surface was subsequently chemical corrosion by erodent (5 g $CuCl_2$ + 100 ml HCl + 100 ml C_2H_5OH) for 30 s, the grain structure could be observed through 9XB-PC optical microscope (OM). The surface was disposed by electrolytic polishing in solution of 20 vol% H_2SO_4 + 80 vol% CH_3OH with 28–30 V input voltage for 3–5 s afterwards. The SUPRA 55 field emission scanning electron microscope (FESEM) was employed to observe γ' phase in detail. After experiencing a fatigue test, the fatigue fracture surface analysis was performed using JEOL-7600F scanning electron microscope (SEM) on cracked specimens after cleaning with ethyl alcohol by ultrasonic cleaner.

The morphology of grain structures and γ' phases of FGH97 is shown in Fig. 1, with an average grain size of 30–40 μm, and the rectangular secondary-precipitated γ' phases closely distribute in the matrix.

2.2 Fatigue crack growth tests

The fatigue crack growth tests were conducted using a servohydraulic test system CMT5204GL in accordance with standard of ASTM E647-81. A resistance-type furnace, attached to the testing machine, was used to heat the specimens. Test temperature fluctuation was maintained within ± 3 °C and the thermal gradient across both the width and length of the specimen did not exceed ± 3 °C. According to the standards of JB/T8189-1999 and ASTM E647-81, standard compact tension (CT) specimen, as shown in Fig. 2, was employed in this study. Each specimen was installed correctly in the displacement-controlling mode, maintained at an appropriate minimum load for elimination of thermal expansion during heating to the

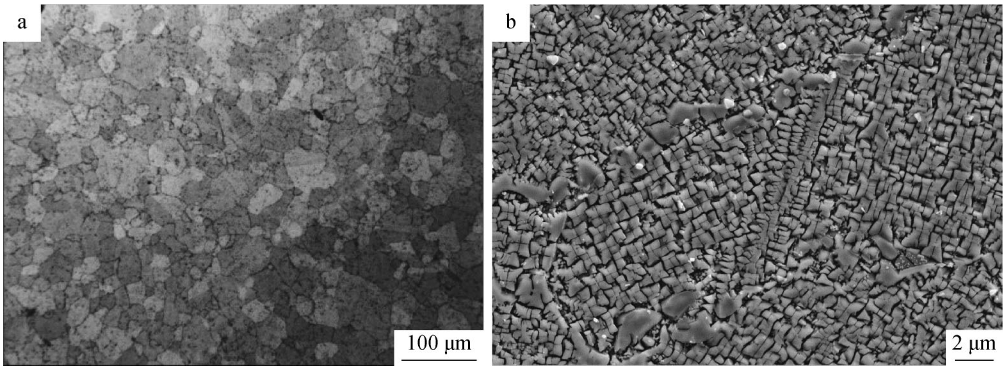

Fig. 1 Microstructures of FGH97 alloy specimen: **a** OM image of grain size and **b** FESEM image of γ' phase

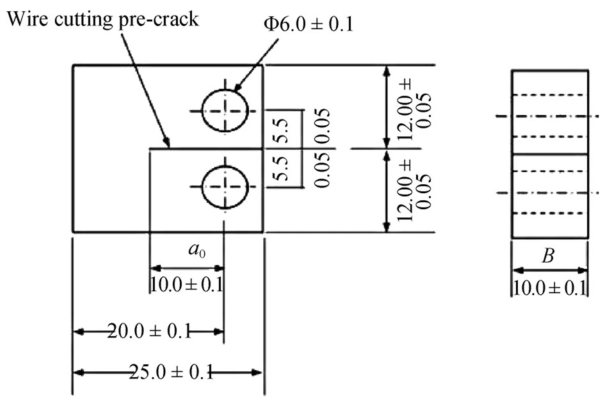

Fig. 2 Schematic illustrations of a CT specimen for crack propagation test, where a_0 representing initial notch length and B representing thickness (unit: mm)

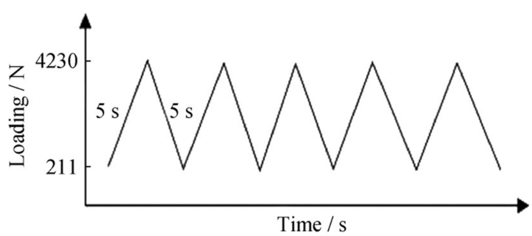

Fig. 3 Fatigue load waveform figure

required test temperature. The tests commenced once the temperature had stabilized in about 30 min.

Studies [20] have shown that FGH97 alloy has excellent properties in 650–750 °C, therefore, three different temperatures 650, 700 and 800 °C were selected to perform fatigue tests in air. A fatigue waveform with 5 s hold-time at the maximum load of 4230 N and 2 s at minimum load of 211 N, and a constant ramp up/ramp down rate yielding 5 s single ramp time each for loading and unloading were applied, as shown in Fig. 3. Tests were conducted under the stress ratio (R, the ratio of applied minimum load to maximum load during a fatigue cycle) of 0.05. A direct-current–potential drop (DCPD) technique was used to continuously monitor the crack propagation during fatigue testing involving the CT specimens, which was a measurement of the overall averaged crack length of the through-cracked area.

2.3 Interruption experiments

To study the mechanism of oxidation and its effects on crack growth at different temperatures, the interruption experiments involving the polished CT specimen were carried out. The experimental processes and parameters were the same as that of the fatigue crack growth tests. The experiment would be stopped once the crack reached a desired length. After cooling to room temperature, the CT specimen was unload and washed by ultrasonic cleaning. It could be further examined by SEM equipped with energy-dispersive spectrometry (EDS) to reveal the path of crack propagation and oxidation at/ahead of the crack tip.

3 Results and discussion

3.1 Fatigue crack propagation behaviors

The experimentally determined points describing the behaviors of crack growth in the investigated FGH97 alloy at different temperatures are presented in the fatigue crack growth life (a–N) curves and FCGR ($\mathrm{d}a/\mathrm{d}N$–ΔK) curves, as shown in Fig. 4, where a, N, and ΔK are crack length, fatigue life and stress intensity factor range, respectively. Note that the latter ones are double logarithmic curves. The FCGR increases as the ΔK value increasing, following the Paris law. It can be seen that the test temperature has a huge effect. With the increase in temperature, fatigue life decreases, and FCGR tends to accelerate. However, in different temperature ranges, the decreasing tendency of fatigue life and the increasing tendency of FCGR have obvious differences. For instance, with temperature rising

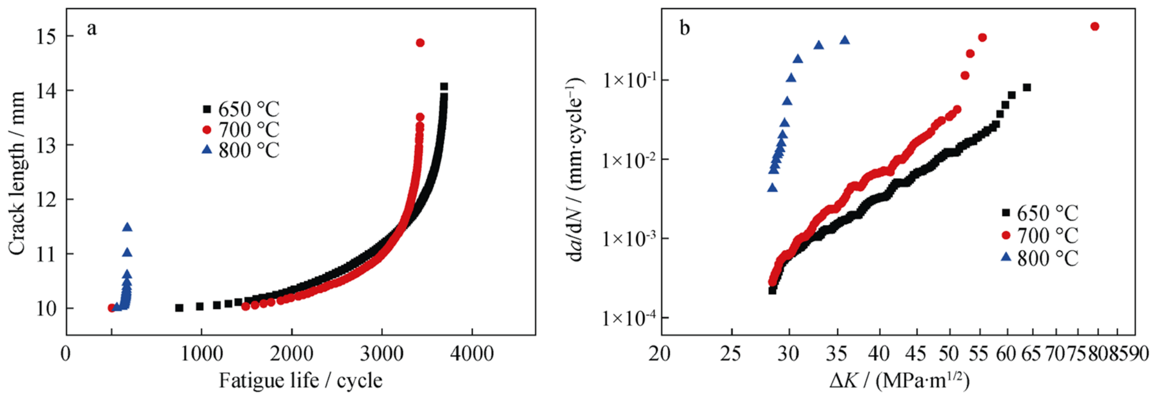

Fig. 4 Lifetime and FCGR curves of FGH97 alloy at different temperatures: **a** $a-N$ and **b** $da/dN-\Delta K$

from 700 to 800 °C, the FCGR is nearly two orders of magnitude higher than that in the range of 650–700 °C.

Considering the fatigue crack growth curves of GH4720Li alloy at various temperatures were conducted by Nai et al. [21], the same phenomenon can be observed, as Fig. 5 shows. The fatigue endurance of the GH4720Li ranging from 700 to 750 °C also has an obvious downward trend, although the experiment condition is different from that of FGH97. It is believed that the rapid degradation of fatigue performance in a certain temperature range is a universal phenomenon for Ni-based superalloys. Therefore, there is a sensitive range of temperature in which the fatigue life decreases sharply, namely, temperature sensitivity.

According to the Paris power law, the FCGR curves consist of three different areas: the near threshold zone, the stabilized extension zone (Paris zone) and the transient zone. Fatigue cycles of the three areas for FGH97 at different temperatures are obtained by taking the derivative of *a-N* curves, as Fig. 6 shows. It is found that the fatigue lives of both the near threshold zone and Paris zone decline dramatically in the temperature-sensitive region, which indicates that the fatigue property for FGH97 alloy is deteriorated.

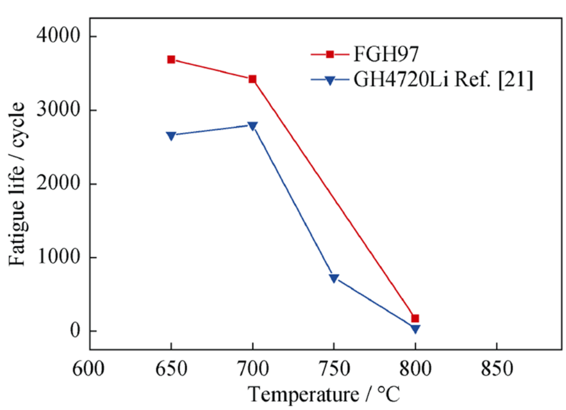

Fig. 5 Lifetime of FGH97 and GH4720Li at different temperatures

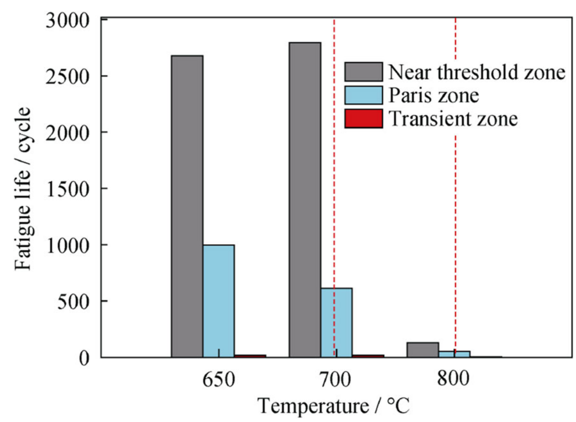

Fig. 6 Lifetime comparison of three stages of FGH97 alloy

If an aero turbo-engine equipped with a FGH97 alloy turbine disk is in service, and operates at the safe application temperature, it will be a longer service life. However, its service life will shorten tremendously when running at the temperature locating in the sensitive range; hence, the safe operation of aircraft or equipment will be threatened seriously. Fatigue performance is a crucial indicator for turbine disk superalloys, which determines whether the alloys can be used for a long time. Therefore, it is necessary to study not only the effects of temperature on fatigue performance for FGH97 alloy, but also the essential reason of temperature sensitivity for fatigue life.

3.2 Microstructural characteristic and mechanical property

In high temperature, changes in microstructures [8] and decay of mechanical properties [9, 22] may lead to the fatigue property of superalloys deterioration. The microstructures of FGH97 specimens experienced fatigue tests under different temperatures are shown in Fig. 7. The size and distribution of γ' phases remain the same, precipitating in rectangle shapes. Although the highest test

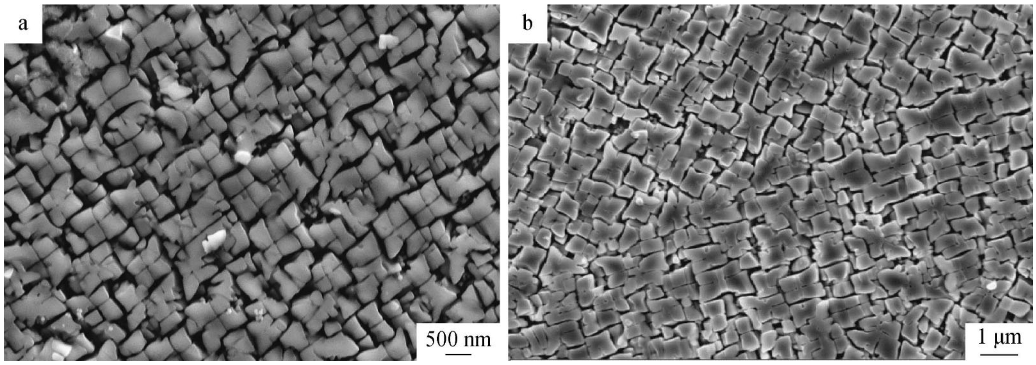

Fig. 7 FESEM images of γ' precipitates for FGH97 after fatigue tests at **a** 650 °C and **b** 800 °C

temperature of 800 °C is very close to the aging temperature of FGH97, the specimen failures instantly in several cycles, therefore the γ' phases do not change in such a short exposure time. Besides, the experimental temperature is much lower than solution temperature and intermediate treatment temperature, therefore, the grain size and grain boundary carbides do not change, too. The phenomenon of decreasing lifetime and increasing FCGR with temperature is unrelated to the microstructures of FGH97 alloy.

When the temperature elevates, mechanical properties related to thermal activation will deteriorate, especially for yield strength (σ_y) and elastic modulus (E) [7, 9]. The decay of them may lead to fatigue property deterioration and FCGR acceleration. In order to study the effects of mechanical properties on FCGR at high temperature, the crack growth curves are modified considering the changes in σ_y and E. Their values of FGH97 [23] at different temperatures are listed in Table 2, indicating that the two items decrease with temperature elevating.

Based on the mentioned method in Ref. [24], the ΔK can be modified as follows: substituting the value of σ_y and E at different temperatures into Eq. (1) and replacing the horizontal ordinate of Fig. 4b with the revised stress intensity factor (ΔK_{norm}), as shown in Fig. 8. It is observed that the three modified curves move slightly to each other; however, such changes can scarcely be identified relative to the vertical intervals of modified curves. Besides, no change can be seen in the slopes of FCGR curves by comparison of the data before and after modification. Thus, the decline of E and σ_y at higher temperature may influence the fatigue

Table 2 Elastic modulus (E) and yield strength (σ_y) of FGH97 alloy at different temperatures

Temperature/°C	E/GPa	σ_y/MPa
650	187.8	897
700	183.9	888
800	176.4	865

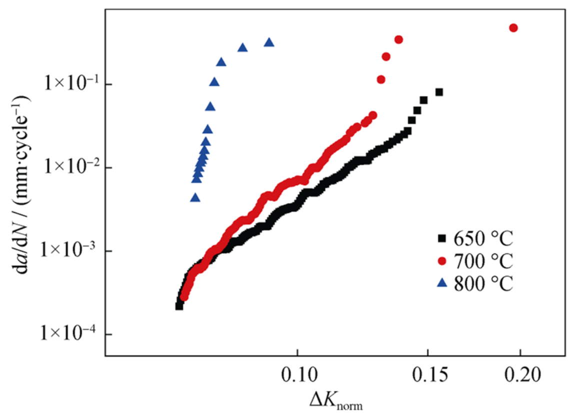

Fig. 8 da/dN−ΔK_{norm} curves of FGH97 alloy after modification of E and σ_y

property of superalloy, but it is not the dominant factor causing the fatigue life to decline sharply in temperature-sensitive region.

$$\Delta K_{\mathrm{norm}} = \Delta K/\sqrt{\sigma_y E}. \quad (1)$$

The FCGR of RR1000 superalloy under fatigue loading (1 s dwell, 0.25 Hz) at temperatures ranged from 550 to 775 °C in vacuum was carried out by Li et al. [25]. With the removal of the oxidizing environment, data of both fast cooling (FC) and slow cooling (SC) RR1000 specimens from all temperatures overlap within a narrow scatter band. It suggests that FCGR is insensitive to temperature within this temperature range investigated in vacuum. On the other hand, it can be seen from overlapping crack growth curves that the recession of mechanical properties (E and σ_y) have little contribution to the enhanced FCGR at elevated temperature.

3.3 Fracture morphology

Appearance of fatigue fracture morphology tested at different temperatures was observed by SEM to analyze the reason that fatigue crack growth behavior decreases with the increase in temperature. Figure 9 presents the fatigue fracture morphologies of FGH97 alloy at 650, 700 and 800 °C, and scale lines of the crack length, fatigue cycles and ΔK values are marked in the images according to fatigue crack propagation curves. Note that the crack growth direction is from bottom to top for all images, as indicated by the red arrow.

Figure 9a illustrates the fracture surface corresponding to the fatigue test at 650 °C, resulting in a transgranular manner. Only a thin oxide layer is observed on the fracture surface. Red mark on the left scale represents the dividing line between the near threshold zone and Paris zone. Even after the Paris area, the crack growth mode is still totally transgranular. This relative smooth fracture surface is associated with the crack traversing individual grains without significant deflection at grain boundaries. Clear radiation ridge and brittle material behavior were characterized by the river pattern within the whole picture. Besides, the secondary cracks are rarely found.

As Fig. 9b shows, at 700 °C, the crack growth of FGH97 is still dominated by transgranular mode, but presents a coarser surface compared with the fracture at 650 °C. In near threshold zone, the fracture surface is smoother, then its roughness increases gradually with crack propagating, accompanied by a handful of secondary cracks, including secondary transgranular cracks and secondary intergranular cracks. Jiang et al. [26] found that the increase in fracture surface roughness was due to the accelerated FCGR. The river pattern and cleavage steps can be observed easily even in Paris zone, so the crack growth mode is still predominantly transgranular in Paris zone.

The fatigue fracture surface at 800 °C presented in Fig. 9c shows a noticeable difference in contrast to the surfaces at lower temperatures. The crack extends along grain boundaries even from the beginning, it is characterized by the intergranular fracture mode, with a tremendously rough surface. Decohesion of grain boundaries is readily apparent, presenting a crystal-sugar-like fracture morphology. Besides, a thick oxide layer and a large number of secondary intergranular cracks can be observed, and some of the secondary cracks are longer and more open. The FCGR is very fast at 800 °C, indicating that the cohesion strength of grain boundaries has been seriously weakened.

According to the fatigue life curves and fracture morphologies of FGH97 at different temperatures, it is found that the sharp reduction of fatigue life in temperature-sensitive region is closely related to the transition of fatigue fracture mode. The relationship between temperature-fatigue life curve and fracture mode is schematically illustrated in Fig. 10, and temperature-sensitive region divides

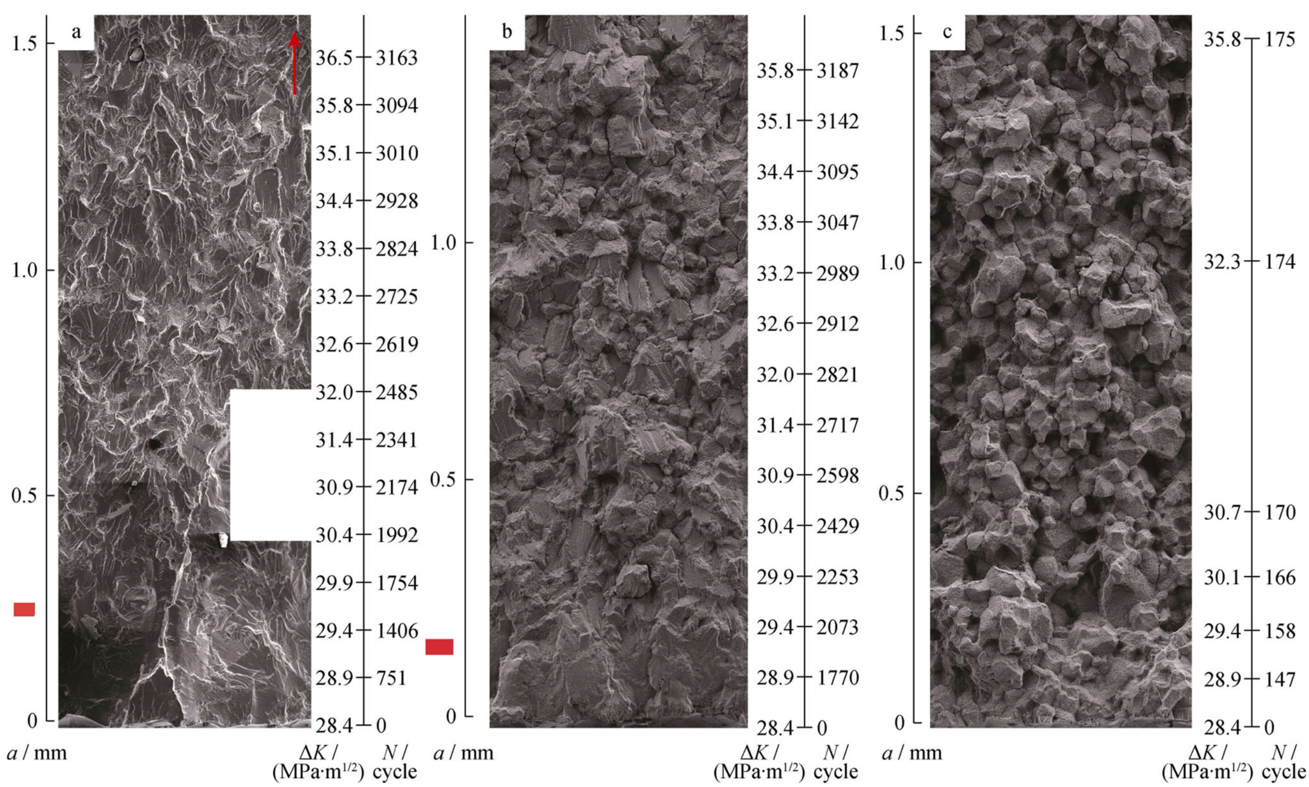

Fig. 9 SEM images for facture morphologies of FGH97 alloy at **a** 650 °C, **b** 700 °C and **c** 800 °C

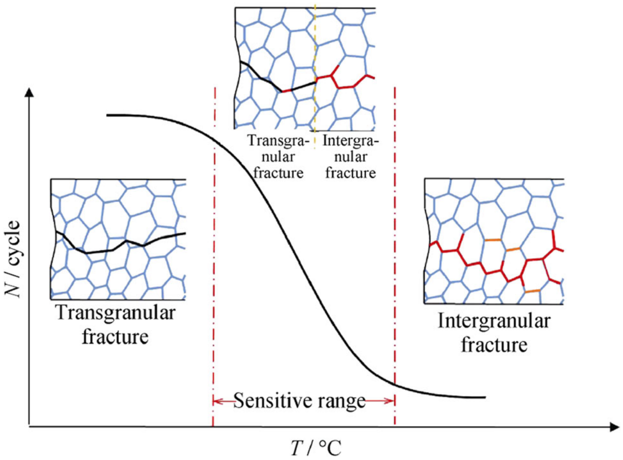

Fig. 10 Diagram of relationship between temperature-cycle and fracture mode

the curve into three sections. FGH97 alloy shows a completely transgranular fracture mode at the temperature that is lower than sensitive region and tends to have a longer fatigue life. When the temperature is higher and on the right side of that region, the fatigue crack advances from the beginning in an intergranular manner, therefore, the FCGR is rapid and fatigue life is very short. However, in the temperature-sensitive region, the fracture mode changes obviously, from transgranular to completely intergranular fracture, accompanied by a sharp decline in fatigue life. It is precisely because of the occurrence of intergranular fracture, the FCGR of FGH97 increases. The difference of microscopic mechanisms of crack growth at various temperatures also account for the differences in slops of FCGR curves.

The abrupt change in fracture mode confirms that the role of oxygen is simply to reduce the grain boundary energy in such a way that the crack growth path becomes intergranular [7]. Grain boundary is the fast channel where the oxygen atoms penetrate the alloy at high temperatures. Oxygen reacts with metal matrix, leading to grain boundary weakness. Therefore, the mechanism responsible for the degraded fatigue performance in temperature-sensitive region may be mainly ascribed to the grain boundary weakness caused by oxidation.

3.4 Research on temperature sensitivity of fatigue life

To study the influences of elevated temperature on fatigue crack propagation, the interruption experiment of FGH97 was carried out at 700 °C. After a cyclic loading for 23 h, namely 2330 cycles, a crack with a length of about 0.8 mm was produced. Morphology of the crack tip is shown in Fig. 11a. The secondary electron micrograph clearly shows that the typical cracking behavior transforms to

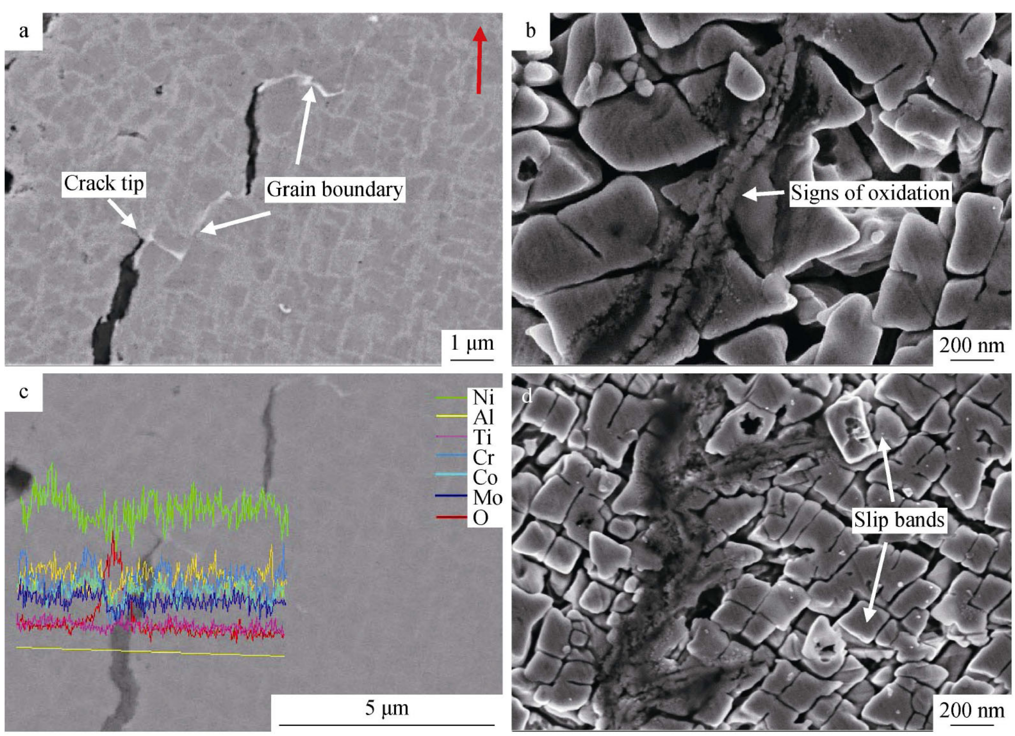

Fig. 11 FESEM images of fatigue crack surface and associated elemental distribution for FGH97 at 700 °C: **a** crack tip advanced along grain boundary and **c** elemental distribution crossing crack by linear scanning of EDS; **b** oxides along flanks of crack and **d** slip bands connected with main crack

intergranular, and a small crack has generated in the front of crack tip, demonstrating that the grain boundary strength is reduced remarkably as well. If the crack continues to propagate, the frontier small crack will be linked with the main crack. In plastic deformation zone, grain boundaries vertical to the loading direction suffer from the greatest normal force during fatigue experiments and tend to produce small cracks firstly [27]. Figure 11b indicates the crack cuts through secondary γ' phase and traces of oxidation along the flanks of crack. Figure 11c is the linear scanning of EDS, showing the enrichment of elemental oxygen around the crack. Despite that distribution of other elements is inaccurate due to the electrolytic etched surface, it is quite clear that amounts of oxide generate at the crack. Oxygen reacts with active elements at the crack tip, producing brittle oxide, cavity, etc. [7, 20], these defects weaken the grain boundary cohesion. In Fig. 11d, oxidation occurs along the slip bands that connect with main crack, and the oxidized slip bands have a length of about 1.7 μm. Beyond the length, oxidation cannot be observed. This manifests that, in plastic deformation area, concentrating stress cause defects and dislocations [28]; therefore, oxygen reacts more violently with active metal elements, the mechanism is similar to the stress assisted grain boundary oxidation (SAGBO).

The interruption experiment for FGH97 at 300 °C (under otherwise identical conditions) was carried out to research the role of high-temperature oxidization on fatigue crack growth and compares its fracture morphology with that at high temperature. Figure 12 shows the feature of crack tip of FGH97 after a cyclic loading up to 202 h (20,000 cycles, 0.7 mm in crack length), the mode of crack growth is quite different to that at 700 °C. In Fig. 12a, the crack extends through connecting slip bands in plastic deformation zone at crack tip, or simply “bridging”, leaving a zig-zag growth path, as shown in Fig. 12b. For most polycrystalline alloys during fatigue, persistent slip bands are regarded as the primary mechanism. As slips accumulate during cyclic loading, plastic deformation manifests into eventual strain localization, leading to crack initiation [28]. In Fig. 12c, d, cracks shear the secondary γ' phase and there is no sign of oxidization around crack, and the crack tip and surrounding grain boundaries are in the same as well.

The fatigue crack growth will not be affected by oxidization, as a result, the crack grows transgranularly and the specimen has a longer fatigue lifetime. However, oxidization will play a vital role in crack growth when the temperature is high enough, due to the reaction rate and penetration depth of oxygen are temperature-dependent.

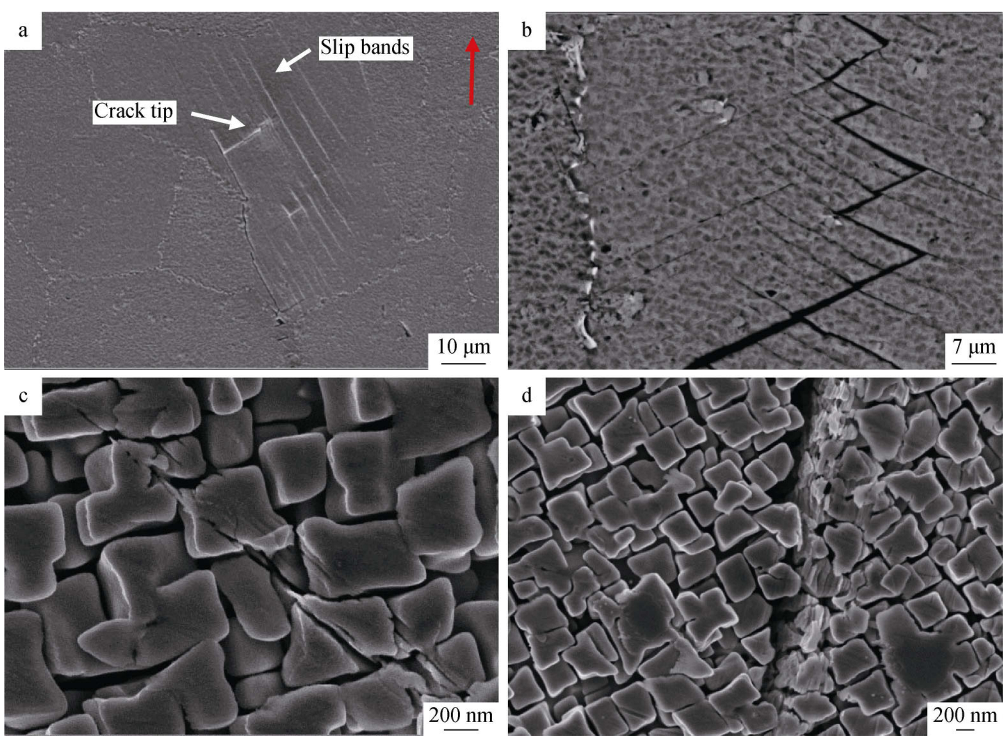

Fig. 12 Microstructure morphologies of fatigue crack surface for FGH97 at 300 °C: **a** SEM image of crack tip and slip bands, **b** SEM image of zig–zag crack extending through connecting slip bands, **c** FESEM image of cracks shearing secondary γ' phase, and **d** FESEM image of no sign of oxidization around crack

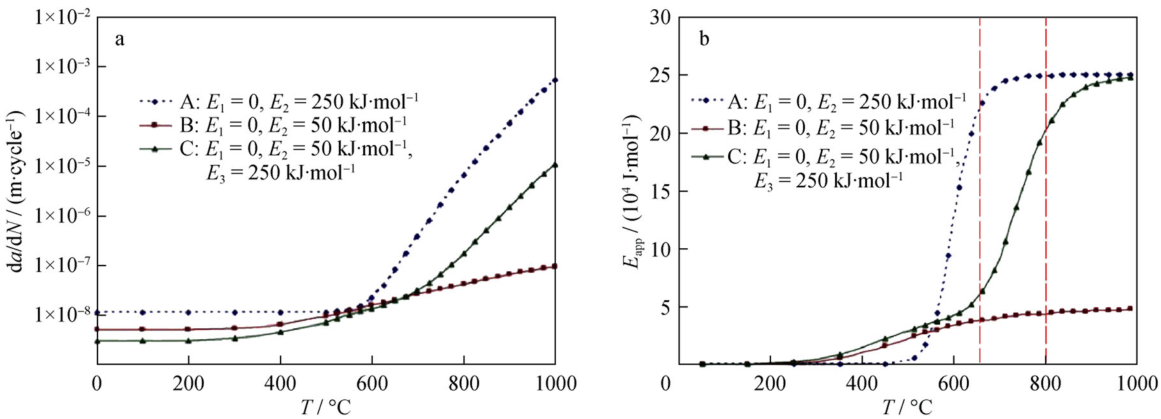

Fig. 13 Fatigue crack growth for three hypothetical scenarios (A, B, C): **a** da/dN and **b** apparent activation energy (E_{app}) (E_1, E_2 and E_3 representing E_{app} for fatigue crack growth at different temperatures, respectively [22])

Oxidization mechanism of superalloys can be identified in terms of oxygen long-range and short-range diffusion processes [29, 30]. At elevated temperature, wedge-shaped oxide [2] generates at crack tip and intrusions into matrix and grain boundaries, resulting in stress concentration at the crack tip and subsequent cavities and microcracks along the oxide/matrix interface or the oxides themselves under cyclic loading, promoting the expansion of cracks. Regarding the later, elemental oxygen can also penetrate the matrix with the assistance of local stress along rapid diffusion paths, such as grain boundary and slip band, leading to the poor recovery of plastic deformation and weak accommodation of strain at the slip band and grain boundary on which oxygen segregates. The two processes mentioned above can also be classified as SAGBO and dynamic embrittlement (DE), respectively [31]. Both of them weaken grain boundary strength [32, 33] and accelerate the FCGR.

The fatigue crack growth behaviors of several of superalloys in vacuum and air have been tested [13, 26, 34]. Experimental results show the FCGR in air is much faster than that in vacuum when other conditions keep the same, which demonstrates that oxygen has enormous influence on crack propagation at high temperature. Oxygen first reacts with active metal elements (Cr, Ti, Al, etc.) at the grain boundaries of crack frontier, therefore crack starts to grow quickly along the reduced grain boundaries. It is usually observed that the FCGR can be 2–3 orders of magnitude higher in the oxidizing environment than that in vacuum or an inert atmosphere [26, 31].

As discussed in detail in Ref. [25], the overlapping FCGR curves of RR1000 alloy suggest that the fatigue property is insensitive to temperature ranged from 550 to 750 °C in vacuum. Both the fracture surfaces are flat transgranular fracture morphology. There is a stark contrast with the FCGR curves of FGH97 alloy (Fig. 4b shows) tested in air in this paper, it can be known clearly that oxidation plays a vital role in degradation of fatigue property at high temperature.

The apparent activation energy (E_{app} [22]) has been applied to analyze the main underlying mechanism causing the progression of the crack under different conditions. Generally, the dominant process for fatigue crack growth is affected by temperature; thus, the measured apparent activation energy depends on temperature. E_{app} is calculated by Eq. (2).

$$E_{app} = \frac{-R_g}{T_2^{-1} - T_1^{-1}} \ln\left(\frac{da}{dN}(T_2) / \frac{da}{dN}(T_1)\right) \tag{2}$$

where R_g is the gas constant (8.31 J·mol^{-1}·K^{-1}), $\frac{da}{dN}(T_2)$ and $\frac{da}{dN}(T_1)$ are FCGR obtained under the same ΔK at two different temperatures ($T_2 > T_1$), respectively.

Among the three hypothetical situations (A, B, C) mentioned to illustrate the issues involved, Case C is most similar to the fatigue failure process in the conditions of high temperature and air environment, as shown in Fig. 13b. Degradation of mechanical performances is the dominant mechanism for fatigue crack growth at 600 °C. However, E_{app} reaches 250 kJ·mol^{-1} and the dominant mechanism switches to oxidization when temperature exceeds 700 °C.

From Case C, it can also be found that there exists a temperature-sensitive region, in which E_{app} increases rapidly and FCGR raises accordingly (Fig. 13a), corresponding to the change of dominant process from degraded mechanical performances to oxidation. E_{app} characterizes the thermal diffusion of particles such as atom and vacancy inside the material and their thermally activated reaction with oxygen. Therefore, the underlying mechanism causing the drastic reduction of fatigue property for FGH97 in sensitive range is the enhanced chemical activity of oxygen and active metal elements around crack tip. Then, the oxidation rate increases and grain boundary strength weakens, which is responsible for the transformation of crack advance from transgranular to intergranular mode and the increase of FCGR.

4 Conclusion

The high-temperature fatigue crack growth behaviors in FGH97 alloy for turbine disk application at different temperatures (650, 700 and 800 °C) in air were investigated using a combination a servohydraulic test system, fractographic and microanalytical investigations. To evaluate the crack propagation mode and oxidation effects, interruption experiments were conducted at 700 and 300 °C, respectively.

There exists a temperature-sensitive region in which the fatigue life drops considerably. In this region, the fatigue fracture converts from a predominantly transgranular mode to an intergranular one, resulting in that the FCGR increases and the lifetime declines rapidly. In air and elevated temperature, oxidation is the dominant factor that gives rise to temperature sensitivity, enhanced thermal activity of oxygen and certain active metal elements accelerate the oxidation reaction. Brittle oxides produced in the place of crack tip and grain boundaries of crack frontier weaken the grain boundary strength, leading to that crack propagates intergranularly, thus degrading the fatigue property of FGH97 dramatically.

Acknowledgements This study was financially supported by the National Natural Science Foundation of China (No. 51371023).

References

[1] Lee SY, Lu YL, Liaw PK, Chen LJ, Thompson SA. Tensile-hold low-cycle-fatigue properties of solid-solution-strengthened superalloys at elevated temperatures. Mater Sci Eng A. 2009; 504(1):64.

[2] Jiang R, Bull DJ, Proprentner D, Shollock B, Reed P. Effects of oxygen-related damage on dwell-fatigue crack propagation in a P/M Ni-based superalloy: from 2D to 3D assessment. Int J Fatigue. 2017;99(1):175.

[3] Ning LK, Zheng Z, An FQ, Tang S, Tong J, Ji HS, Yu HW. Thermal fatigue behavior of K125L superalloy. Rare Met. 2016; 35(2):172.

[4] Kitaguchi HS, Li HY, Evans HE, Ding RG, Jones IP. Oxidation ahead of a crack tip in an advanced Ni-based superalloy. Acta Mater. 2013;61(6):1968.

[5] Li SL, Qi HY, Yang XG. Oxidation-induced damage of an uncoated and coated nickel-based superalloy under simulated gas environment. Rare Met. 2018;37(3):204.

[6] Han ZX. Effects of temperature on thermal fatigue properties of some wrought superalloys. Gas Turbine Exp Res. 2007;20(4):53.

[7] Osinkolu GA, Onofrio G, Marchionni M. Fatigue crack growth in polycrystalline IN718 superalloy. Mater Sci Eng A. 2003; 356(1–2):425.

[8] Kruml T, Obrtlik K. Microstructure degradation in high temperature fatigue of TiAl alloy. Int J Fatigue. 2014;65:28.

[9] Krupp U, Kane WM, Liu X, Dueber O, Laird C. The effect of grain-boundary-engineering-type processing on oxygen-induced cracking of IN718. Mater Sci Eng A. 2003;349(1–2):213.

[10] Calvarin-Amiri G, Huntz AM, Molins R. Effect of an applied stress on the growth kinetics of oxide scales formed on Ni-20Cr alloys. Mater High Temp. 2001;18(2):91.

[11] Miller CF, Simmons GW, Wei RP. Evidence for internal oxidation during oxygen enhanced crack growth in P/M Ni-based superalloys. Scr Mater. 2003;48(1):103.

[12] Tong J, Dalby S, Byrne J. Crack growth in a new nickel-based superalloy at elevated temperature—part III: characterisation. J Mater Sci. 2005;40(5):1229.

[13] Pfaendtner JA Jr, McMahon CJ. Oxygen-induced intergranular cracking of a Ni-base alloy at elevated temperatures—an example of dynamic embrittlement. Acta Mater. 2001;49(16): 3369.

[14] Krupp U, Kane WM, Laird C, Mcmahon CJ. Brittle intergranular fracture of a Ni-base superalloy at high temperatures by dynamic embrittlement. Mater Sci Eng A. 2004;387(1):409.

[15] Karabela A, Zhao LG, Lin B, Tong J, Hardy MC. Oxygen diffusion and crack growth for a nickel-based superalloy under fatigue-oxidation conditions. Mater Sci Eng A. 2013;567(8):46.

[16] Zheng XL. On some basic problems of fatigue research in engineering. Int J Fatigue. 2001;23(9):751.

[17] Liu H, Bao R, Zhang JY, Fei BJ. A creep-fatigue crack growth model containing temperature and interactive effects. Int J Fatigue. 2014;59(3):34.

[18] Zhong Z, Gu Y, Yuan Y, Cui C, Yokokawa T. Fatigue crack growth behavior of a newly developed Ni-Co-base superalloy TMW-2 at elevated temperatures. Mater Sci Eng A. 2012; 552(34):464.

[19] Nai QL, Dong JX, Zhang MC, Zheng L, Yao ZH. Fatigue behavior of powder metallurgy superalloy FGH97. Chin J Eng. 2016;28(2):248.

[20] Zhang YW, Shangguan YH. Research and development in P/M superalloy. Power Metall Ind. 2004;14(6):30.

[21] Nai QL, Dong JX, Zhang MC, Zheng L, Yao ZH, Qu JL. Temperature sensitivity of fatigue crack growth rate for GH4720Li alloy. Rare Metal Mater Eng. 2017;46(10):2915.

[22] Starink MJ, Reed PAS. Thermal activation of fatigue crack growth: analysing the mechanisms of fatigue crack propagation in superalloys. Mater Sci Eng A. 2008;491(1–2):279.

[23] CSM. China Superalloys Handbook. Beijing: China Zhijian Publishing House; 2012. 878.

[24] Lou J, Mercer C, Soboyejo WO. An investigation of the effects of temperature on fatigue crack growth in a cast lamellar Ti-45Al-2Mn-2Nb+0.8 vol.% TiB_2 alloy. Mater Sci Eng A. 2001;319(1):618.

[25] Li HY, Sun JF, Hardy MC, Evans HE, Williams SJ. Effects of microstructure on high temperature dwell fatigue crack growth in a coarse grain P/M nickel based superalloy. Acta Mater. 2015; 90:355.

[26] Jiang R, Everitt S, Lewandowski M, Gao N, Reed PAS. Grain size effects in a Ni-based turbine disc alloy in the time and cycle dependent crack growth regimes. Int J Fatigue. 2014;62(30):217.

[27] Lillo T, Cole J, Frary M, Schlegel S. Influence of grain boundary character on creep void formation in alloy 617. Metall Mater Trans A. 2009;40(12):2803.

[28] Sangid MD. The physics of fatigue crack initiation. Int J Fatigue. 2013;57(12):58.

[29] Prakash DGL, Walsh MJ, Maclachlan D, Korsunsky AM. Crack growth micro-mechanisms in the IN718 alloy under the combined influence of fatigue, creep and oxidation. Int J Fatigue. 2009;31(11):1966.

[30] Ghonem H, Nicholas T, Pineau A. Elevated temperature fatigue crack growth in alloy 718—part II: effects of environmental and material variables. Fatigue Fract Eng Mater Struct. 2010;16(6):577.
[31] Jiang R, Reed PAS. Critical assessment 21: oxygen-assisted fatigue crack propagation in turbine disc superalloys. Mater Sci Technol. 2016;32(5):401.
[32] Evans HE, Li HY, Bowen P. A mechanism for stress-aided grain boundary oxidation ahead of cracks. Scr Mater. 2013;69(2):179.
[33] Andrieu E, Molins R, Ghonem H, Pineau A. Intergranular crack tip oxidation mechanism in a nickel-based superalloy. Mater Sci Eng A. 1992;154(1):21.
[34] Molins R, Hochstetter G, Chassaigne JC, Andrieu E. Oxidation effects on the fatigue crack growth behaviour of alloy 718 at high temperature. Acta Mater. 1997;45(2):663.