Detection and analysis of high temperature fatigue fracture surface oxygen diffusion of GH4133B superalloy used in turbine disk of aero-engine

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Abstract. The chemical contents on the fatigue fracture surface of GH4133B superalloy piece suffering tension-tension fatigue and operated at high temperature 600 °C is analyzed using an energy dispersive spectrometer, and the element content percentages at the position of carbide inclusion and that of second-phase particle in the fatigue source region are measured. It is shown that under the conditions of high temperature and air environment, the carbon element in the carbide inclusion can be easily oxidized, which results in accelerating fatigue crack nucleation and microcrack generation, reducing the fatigue crack initiation lifetime. The oxygen concentration is detected on the fatigue fracture surface along the crack propagation direction, the oxygen diffusion model is constructed, and the oxygen concentration gradient equation is derived. It is found that the oxygen content decreases gradually along the crack propagation direction, suggesting that at the crack initiation and short crack propagation stage, the alloy element reacts sufficiently with oxygen for a long time, and the oxidation accelerates the crack initiation, while at the long crack growth stage, the crack propagation rate is faster, so the alloying elements do not have adequate time for reacting with oxygen, resulting in the low oxygen content, and the oxidation has a small influence on the long crack propagation rate.

1. Introduction

As far as the studies of the analysis of fatigue fracture surface oxygen diffusion of nickel-base superalloy operated at elevated temperature in air environment are considered, some research works have been done. Prakash [1] reviewed the crack growth micromechanisms in IN718 nickel-base superalloy under the combination effect of fatigue with creep and oxidation, and studied the different fatigue crack growth phenomena in IN718 superalloy. Applying different dwell times at the maximum stress, Hu [2] conducted the creep-fatigue tests for GH720Li nickel-based superalloy at high temperature of 650 °C with a stress ratio of 0.1, and discussed the microscopic mechanism of the creep-fatigue behavior of GH720Li superalloy by SEM with EDS on the fracture surfaces, and found that increasing dwell time leads to a changing from more fatigue failure to dominated creep failure. Under typical loading waveforms at
selected loading frequencies, Tong [3] investigated the role of creep, fatigue and oxidation in crack growth of two powder metallurgy (PM) nickel-base superalloys, and utilized a scanning electron microscopy to identify the fracture mode and to facilitate the oxidation evaluation, and found that oxidation appears to be the predominant mechanism for crack growth under static and long dwell loading conditions. Zhang [4] studied the microstructure of the nickel-base superalloys at an elevated temperature of 1000°C, and observed both the evaporation and reconstruction of alloying elements for the first time with the atomic resolution at high temperature. Ruiz-Sabariego [5] studied the oxidation assisted fatigue crack growth under complex non-isothermal loading conditions in an N18 nickel-base superalloy.

In this paper, the chemical content on the high temperature fatigue fracture surface of GH4133B superalloy specimen is analyzed using an energy dispersive spectrometer, and the element content percentages at the position of carbide inclusion and that of second-particle in the fatigue source region are measured. The oxygen concentration is detected on the fatigue fracture surface along the direction of fatigue crack propagation, and the oxygen concentration gradient equation is derived.

2. Experimental

2.1. Materials and specimens
The material used in this research is a powder metallurgy nickel-base superalloy GH4133B, which is a modified superalloy made by adding an appropriate amount of Mg and Zr microalloying based on GH4133 superalloy, to eliminate the notch sensitivity of GH4133 superalloy above 750 ºC, resulting in an increasing of the service lifetime and a substantial increasing of the rupture strength and ductility.

The chemical compositions of GH4133B superalloy in weight percentage are shown in Table 1. The specimens are machined from a turbine disk. The heat treatment process is as follow: 1080±10 ºC, 8 hours, air cooling with 750±10 ºC, 16 hours air cooling. The specimen is machined in the shape of sheet specimen with length of 50 mm, width of 15mm, thickness of 3 mm, effective length of 24 mm, effective width 5 mm, and a transition arc with a radius of 3 mm.

| Element | Ni  | C   | Cr  | Al  | Ti  | Fe  | Nb   |
|---------|-----|-----|-----|-----|-----|-----|------|
| wt. %   | ≥69.3607 | ≤0.06 | 19.00–22.00 | 0.75–1.15 | 2.50–3.00 | ≤1.5 | 1.30–1.70 |

| Element | Mg  | Zr  | B   | Ce  | Mn  | Si  | P   |
|---------|-----|-----|-----|-----|-----|-----|-----|
| wt. %   | 0.001–0.01 | 0.01–0.1 | ≤0.01 | ≤0.01 | ≤0.35 | ≤0.65 | ≤0.015 |

| Element | S   | Cu  | Bi  | Sn  | Sb  | Pb  | As  |
|---------|-----|-----|-----|-----|-----|-----|-----|
| wt. %   | ≤0.007 | ≤0.07 | ≤0.0001 | ≤0.0012 | ≤0.0025 | ≤0.001 | ≤0.0025 |

2.2. High temperature fatigue tests
At elevated temperature of 650 ºC, the fatigue tests for the specimens of GH4133B superalloy are conducted on an MTS809 materials testing machine at various stress ratios, maximum stress levels and in atmospheric environment. A sine wave with a frequency of 2 Hz is adopted in the tests. These tests are divided into two groups. For the first group, the maximum stress $\sigma_{max}$ is set as a fixed value of 700 MPa, the stress ratio $R$ ranges from 0.01, 0.1, 0.2 to 0.4, the specimens are numbered from S1 to S4, and the fatigue lifetime is measured as 27903, 26823, 27982 and 42659 cycles, respectively. For the second group, the stress ratio is fixed as a constant of 0.1, the maximum stress level varies from 900 MPa, 800 MPa, 600 MPa to 550 MPa, the specimens are numbered from S5 to S8, and the fatigue lifetime is individually recorded as 3449, 10976, 54769 and 178117 cycles.

The fracture surface morphologies on the fatigue failure fractography are investigated using a JSM-6360LV scanning electron microscopy (SEM), and the chemical compositions on fracture surface of GH4133B superalloy specimen are analyzed using an energy dispersive spectrometer (EDS) that is installed on the JSM-6360LV.
3. Element content and oxygen diffusion analysis

3.1. Element content analysis

For the specimen operated at stress ratio of 0.01 with a fixed maximum stress level of 700 MPa, the positions of carbide inclusions in the crack initiation region for element content percentage analysis on the fracture surface are shown in Figure 1(a) and (b), the element content energy spectrum diagrams at detected positions are shown in Figure 2(a) and (b), and the element contents of wt.% and at.% at Spectrum 1 and 2 on the fracture surface in the crack initiation region are listed in Table 2.

![Figure 1.](image1.png)

![Figure 2.](image2.png)

**Figure 1.** The positions of carbide inclusions or second-phase particles in the crack initiation region of GH4133B superalloy at $R=0.01$ and $\sigma_{\text{max}}=700$ MPa. (a) Spectrum 1; (b) Spectrum 2.

**Figure 2.** The element content energy spectrum diagrams in the crack initiation region of GH4133B superalloy at $R=0.01$ and $\sigma_{\text{max}}=700$ MPa. (a) Spectrum 1; (b) Spectrum 2.

**Table 2.** The element contents of wt.% and at% at Spectrum 1 and 2 in the crack initiation region.

| Element | Spectrum 1 wt.% | Spectrum 1 at.% | Spectrum 2 wt.% | Spectrum 2 at.% | Element | Spectrum 1 wt.% | Spectrum 1 at.% | Spectrum 2 wt.% | Spectrum 2 at.% |
|---------|-----------------|-----------------|-----------------|-----------------|---------|-----------------|-----------------|-----------------|-----------------|
| C       | 54.61           | 68.23           | 53.56           | 67.04           | Cr      | 1.16            | 0.29            | 2.70            | 0.71            |
| O       | 36.46           | 29.97           | 35.55           | 30.53           | Zr      | 2.47            | 0.36            | 4.92            | 1.15            |
| Mg      | 0.29            | 0.16            | 0.01            | 0.01            | Nb      | 1.80            | 0.25            | 1.57            | 0.24            |
| Al      | 0.04            | 0.02            | 0.20            | 0.10            | Ce      | 0.20            | 0.02            | 1.09            | 0.16            |
| Ti      | 0.72            | 0.20            | 0.16            | 0.04            | Ni      | 2.25            | 0.50            | 0.24            | 0.02            |

It is seen from Figure 1 and Table 2 that comparing with the chemical compositions of GH4133B superalloy listed in Table 1, the carbon content in carbide inclusion is much higher than the average value in GH4133B superalloy. Under the condition of cyclic loading, some microcracks are produced by the separation of carbide inclusions from the matrix material, which results in the initiation of fatigue cracks. In high temperature environment, carbon is easy to oxidize. This can be found from oxygen content at Spectrum 1 and 2 in Table 2, which is much higher than the average oxygen content of GH4133B superalloy. The oxidation of carbon elements accelerates the nucleation of fatigue crack,
and further reduces the fatigue crack initiation lifetime of GH4133B superalloy. Simultaneously, the stress concentration occurring at carbide inclusions may also accelerate the fatigue crack initiation.

For the specimen operated at various stress ratios and at a fixed maximum stress of 700 MPa, the detected point positions in the crack initiation region of GH4133B superalloy are shown in Figure 3, the corresponding element content energy spectrum diagrams at detected positions are shown in Figure 4, and the element contents of wt.% and at.% at the points Spectrum 3, Spectrum 4, Spectrum 5 and Spectrum 6 on the fracture surface in the fatigue crack initiation region are listed in Table 3.

Figure 3. The positions of detected points in the crack initiation region of GH4133B superalloy at various stress ratios and at $\sigma_{\text{max}}=700$ MPa. (a) $R=0.01$; (b) $R=0.1$; (c) $R=0.2$; (d) $R=0.4$.

Figure 4. The element content energy spectrum diagrams of GH4133B superalloy at various stress ratios and at $\sigma_{\text{max}}=700$ MPa. (a) $R=0.01$; (b) $R=0.1$; (c) $R=0.2$; (d) $R=0.4$. 
Table 3. The element contents of wt.% and at.% at detected points in the crack initiation region.

| Element | Spectrum 3 | Spectrum 4 | Spectrum 5 | Spectrum 6 |
|---------|------------|------------|------------|------------|
|         | wt.%       | at.%       | wt.%       | at.%       | wt.%       | at.%       | wt.%       | at.%       |
| C       | 10.87      | 29.98      | 7.08       | 24.19      | 11.38      | 35.81      | 12.82      | 36.47      |
| O       | 11.44      | 23.70      | 4.44       | 11.39      | 2.48       | 5.87       | 6.44       | 13.75      |
| Mg      | 0.51       | 0.70       | 0.30       | 0.51       | 0.36       | 0.56       | 0.48       | 0.68       |
| Al      | 1.00       | 1.22       | 0.43       | 0.65       | 0.66       | 0.93       | 0.94       | 1.19       |
| Ti      | 2.37       | 1.64       | 2.57       | 2.21       | 2.59       | 2.04       | 2.87       | 2.04       |
| Cr      | 15.99      | 10.19      | 18.44      | 14.56      | 18.40      | 13.37      | 17.91      | 11.77      |
| Zr      | 0.29       | 0.10       | 0.19       | 0.08       | 0.32       | 0.13       | 0.04       | 0.01       |
| Nb      | 1.16       | 0.42       | 1.15       | 0.51       | 1.31       | 0.53       | 1.91       | 0.70       |
| Ce      | 0.37       | 0.09       | 0.17       | 0.05       | 1.01       | 0.27       | 1.31       | 0.32       |
| Ni      | 56.00      | 32.96      | 65.23      | 45.85      | 61.49      | 40.49      | 55.28      | 33.07      |

It is seen from Figure 3 and Table 3 that comparing with the chemical compositions listed in Table 1, the oxygen content in the crack initiation region is much higher than the mean value in GH4133B superalloy. In the air environment at high temperature, the chemical property of oxygen is very active, the metal and non-metallic elements in the fatigue source region are easy to react with oxygen, which result in that these elements are oxidized, leading to an increasing of oxygen content in the fatigue source region. On one hand, the oxidation of metallic and non-metallic elements in the alloy prevents the further oxidation of other metallic or non-metallic elements, showing an effect of passivation crack tip and shielding crack propagation. On the other hand, the oxidation of metallic and non-metallic elements in the alloy makes the crack tip sharpening, and showing an effect of accelerated fatigue crack initiation and propagation. Therefore, at the stage of fatigue crack initiation and short crack propagation, there are two competitive mechanisms of accelerating and decelerating crack growth for the oxidation of metal and non-metallic elements. On the macroscopic view, the crack growth rate in the short crack growth zone shows a fluctuation of accelerated and decelerated propagation. For the specimen operated at various maximum stress levels ranging from 900 MPa to 550 MPa at a fixed stress ratio of 0.1, similar results are found for the oxygen detection in the crack source zone.

3.2. Oxygen diffusion analysis

For the specimen carried out at stress ratio of 0.01 and at a maximum stress level of 700 MPa, the detection of oxygen element content on the fracture surface along the crack propagation direction in the fatigue source region is carried out by using an energy dispersive spectrometer, and the detection path is shown in Figure 5. The experimental data of oxygen content in wt.% along the crack propagation direction are shown in Figure 6. It can be seen from Figure 6 that the oxygen content decreases gradually on the whole along the crack propagation in the fatigue source region, indicating that the sooner the early crack is, the metallic and non-metallic elements on the crack surface have more time to react with oxygen, leading to a higher oxygen content percentage in the crack initiation region. While for the later stage of crack growth, the crack growth rate is faster, and the metallic and non-metallic elements in the long crack growth region have no adequate time to react with oxygen, resulting in a lower oxygen content percentage in the long crack growth region. Therefore, oxidation has no significant acceleration effect on long crack propagation.

Analyzing the experimental data of oxygen content, it can be found that although these data have some degree of scattering, the oxygen content decreases exponentially along with the crack length. The relation expression between the oxygen content $C_{\text{oxygen}}$ and the crack length $l_{\text{crack}}$ is written as

$$C_{\text{oxygen}} = \exp(A + Bl_{\text{crack}})$$  \hspace{1cm} (1)

where, $A$ and $B$ are the material parameters. The gradient equation of oxygen element concentration $(dC_{\text{oxygen}}/dl_{\text{crack}})$ along the crack propagation direction is derived as
\[ \frac{dC_{\text{oxygen}}}{dl_{\text{crack}}} = B \exp(A + Bl_{\text{crack}}) \]  

According to Equation (1), the experimental data of oxygen element content is analyzed using nonlinear regression analysis method, and the values of material parameters \( A \) and \( B \) are identified as \( 2.78540 \) and \( -0.00802 \) mm\(^{-1}\). That is, the oxygen element diffusion equation of the oxygen content \( C_{\text{oxygen}} \) along the crack length \( l_{\text{crack}} \) for GH4133B superalloy operated at high temperature 650 ºC is

\[ C_{\text{oxygen}} = \exp(2.78540 - 0.00802l_{\text{crack}}) \]  

And the corresponding gradient equation of oxygen element content along the crack length is

\[ \frac{dC_{\text{oxygen}}}{dl_{\text{crack}}} = -0.00802\exp(2.78540 - 0.00802l_{\text{crack}}) \]  

The comparison between the theoretical curve and experimental data is shown in Figure 6. It can be found from Figure 6 that the predicted results agree with experimental data.

![Figure 5. Detection path of oxygen content.](image1)

![Figure 6. The wt.% of oxygen along crack.](image2)

4. Conclusions

The conclusions are given as following:

(a) Under high temperature and air environment, the carbide inclusions in GH4133B superalloy are easily oxidized, which results in accelerating fatigue crack nucleation and microcrack generation, reducing the fatigue crack initiation lifetime.

(b) At the positions of the carbide inclusions and other second-phase particles, the oxygen contents on the fracture surface in the fatigue source region are dramatically higher than the average oxygen content in the GH4133B superalloy.

(c) The oxygen concentration is detected on the fatigue fracture surface along the fatigue crack propagation direction. It is found that the oxygen element content on the fatigue fracture surface of GH4133B superalloy decreases exponentially along with the crack length.

Acknowledgements

The authors gratefully acknowledge the financial support of the Research Foundation of Education Bureau of Hunan Province, (No.14A144).

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