Mechanism of Low Cycle Fatigue Damage for Nickel-Based Powder Superalloy

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Abstract. In this paper, the in-situ scanning electron microscope (SEM) was used to study crack initiation and propagation during the fatigue process of nickel-based superalloys prepared by powder metallurgy at room temperature (RT) and high temperature (HT). It could be found that the crack initiation of alloy is mainly along the <111> direction of crystallographic orientation at room temperature, cracks are parallel, perpendicular, at an angle of 53° and 60°. Cracks initiated in the crystal propagate in zigzag manner at an angle to each other, the direction of growth is perpendicular to the direction of stress. The fatigue cycle required for crack initiation at the grain boundary is much lower than that in the crystal, and the crack propagation is much faster. At initial stage, cracks expand along the grain boundary to a certain extent and then propagate in a transgranular manner, the direction of which is approximately perpendicular to the stress direction. At high temperature, the fatigue crack initiation time is much longer than the room temperature, and the cycle times required for crack initiation at the same stress ratio are 15 times higher than that at room temperature. The time and length of fatigue crack propagation along the grain boundary are larger than those at room temperature, but the length of the transgranular expansion is slightly smaller.

1. Introduction

Because of the characteristics of small grain size, uniform structure, high strength and good fatigue resistance, nickel-based power superalloy have become the material of choice for key parts such as aero-engine turbine discs [1, 2]. Fang Bin, Wang Mengya, Tian Gaofeng and other researchers [3-8] conducted in-depth research on the microstructure, mechanical properties, constitutive equation, recrystallization equation, processing map, grain boundary engineering of nickel-based powder superalloys, but further research is needed on the fatigue life and its influencing factors in service under harsh environment (high temperature, high load stress) [9]. For superalloy parts, fatigue failure is the most common failure mode. It occurs from crack initiation, propagation and fracture. It is characterized by no obvious plastic deformation before failure, so fatigue fracture usually occurs suddenly [10-12]. Fatigue-induced fracture accounts for 70%~80% of engineering component or structure fracture accidents.

The in-situ test refers to the application of a servo fatigue testing machine with a scanning electron microscope (SEM) for fatigue testing. The advantage of this method is that the initiation and early propagation of small fatigue cracks can be observed in real time, which provides experimental supports for the study of the mechanism of the initiation and propagation of fatigue cracks. This kind of research is very important for improving material preparation technology and fatigue life of...
materials [13]. Liang Yan et al. [14] studied fatigue crack initiation and propagation behavior of 2524 aluminum alloy with high strength at room temperature by using in situ SEM fatigue experience. It can be found that the fatigue crack of 2524 alloy nucleates on the interface between the inclusion particles or the inclusion particles and the matrix material. The main crack propagates along the slip zone in the shear mechanism or the direction of the microcrack before the crack tip, and the propagation direction and velocity of microcrack propagation is affected by the grain boundaries. Xie Xishan et al. [15] in-situ analysis of fatigue fracture caused by inclusions in nickel-based powder superalloys, the results show that fatigue cracks are easy to initiate and propagate into the matrix at the inclusion/substrate interface and inclusions.

At present, high reliability is proposed for nickel-based powder superalloys as aero-engine vortex, and low cycle fatigue performance is a key factor in its service life. In this paper, the initiation and propagation of cracks in nickel-based superalloys during low-cycle fatigue have been studied by in-situ fatigue experiments at RT and HT.

2. Experimental materials and methods

2.1. Materials
The samples of nickel-based superalloys used in this paper are prepared by powder metallurgy (PM) method. The composition is shown in Table 1.

|   | Ni  | Cr  | Co  | Mo  | Al  | Ti  | W   | Nb  | C   | B   | Zr  |
|---|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Bal. | 15.78 | 13.04 | 4.33 | 4.14 | 3.88 | 2.26 | 0.82 | 0.03 | 0.01 | 0.03 |

2.2. Mechanical Properties Test
The mechanical properties of the samples at room temperature, 400°C and 650°C were measured by Universal Capacitance Testing Machine. The stress and strain values of the experimental process were converted by the extensometer and the load.

2.3. In-situ SEM Fatigue Test
In-situ fatigue test was used to characterize the fatigue properties of the samples. The alloy sample was mechanically cut into the in-situ fatigue sample shown in Figure 1. In order to ensure the purpose of the experimental observation, a gap was formed at the narrowest point in the middle of the sample, and the notch effect was obtained here, and the initiation of the crack was more easily observed. The sample was sanded to the upper and lower surfaces and side to 2000# and polished, the sample size was accurately measured for stress conversion. The stress was calculated from the thinnest part of the middle of the sample and rounded down. The stress was not adjusted during the experiment. To show the change in tissue during the in-situ stretching of the sample, the sample was subjected to mild chemical attack prior to stretching to enhance the contrast.

For the high temperature fatigue test, in order to prevent the sample from oxidizing during the high temperature experiment, the whole process was carried out in a low vacuum environment. The experimental results were observed for the first time as cracks appearing as crack initiation cycles.

![Figure 1. Samples for in situ fatigue test](image-url)
2.4. Microstructure Analysis

The microscopic morphology of the sample was analyzed using a metallographic microscope and CX-200TA SEM equipped with a mass spectrometer.

3. Results and Discussion

3.1. Mechanical Properties

![Stress-strain curve of nickel-based alloy](image)

**Figure 2.** Stress-strain curve of nickel-based alloy

| T/°C | σ_s/MPa | σ_b/MPa | A/% |
|------|---------|---------|-----|
| R.T. | 1200    | 1650    | 30% |
| 400  | 1100    | 1500    | 17% |
| 650  | 850     | 1400    | 20% |

Table 2. Mechanical properties of nickel-based alloy.

Fig.2 and Table.2 represents the RT and elevated temperature stress-strain curves and mechanical properties of nickel-based alloys. The mechanical properties are as follows: the yield strength at R.T. is 1200 MPa, the UTS is 1650 MPa, and the elongation after fracture is about 30%. At 400 °C, the yield stress was reduced to 1100 MPa, the tensile strength was reduced to approximately 1500 MPa, and the elongation after fracture was approximately 17%. At 650 °C, the alloy had no obvious yield limit. Using the equivalent calculation of 0.2% deformation, it can be considered that the yield strength is 850 MPa, the tensile strength was reduced to about 1400 MPa, and the elongation after fracture was about 20%

3.2. Fatigue at RT

![In-situ SEM fatigue morphology of alloy at room temperature and 1150Mpa load stress](image)

**Figure 3.** In-suit SEM fatigue morphology of alloy at room temperature and 1150Mpa load stress

The in-situ fatigue test in RT used a load of 800 N, 10 Hz frequency and 0.1 stress ratio; the sample thickness was 0.81 mm, the narrowest part of the width was 0.86 mm, and the load stress was about 1150 MPa. The sample eventually fractured when cycled to 6636 cycles. The process of high-stress in situ experiment at room temperature is shown in FIG. 3.
It can be found in Fig. 3(b) that crack initiation occurred on the surface of the sample when it was cycled to about 727 cycles, most of the cracks were initiated in the crystal and extended along a certain crystallographic orientation, with a few cracks passed through the grain boundaries. The cracks were parallel, perpendicular or at a specific angle to each other. The geometric relationship between the cracks in the image was analyzed using ImageJ software. It can be seen from the figure 3(b) that there were mainly four orientation relationships between the cracks: parallel, vertical, 60° angle and 53° angle. Since the main component of the alloy was Ni, the crystal structure was face centered cubic (FCC). The main slip system that was activated in the FCC structure was the <111> direction, and the projection angle of the <111> direction in each direction could be calculated by crystallography, the result is shown in Fig.4. It can be seen that the projection angle of the <111> direction in the <001> direction was 90°, the projection angle in the <011> direction was 53°, and the projection angle in the <111> direction was 60°. These angles were consistent with the angles observed in Figure 3(b). Therefore, it can be judged that the crack was preferentially initiated in the crystal along the starting slip system.

![Figure 4. Projection angle of <111> direction in FCC structure](image1)

![Figure 5. Angel between crack direction and stress direction](image2)

Statistical methods were used to analyze the angle between the crack initiation direction and the stress direction. Fig.5 shows that the angle between the crack and the stress direction was mainly concentrated in the range of 80°~90° and 30°~40°. This is mainly because the tensile stress was the largest when the crack was approximately 80° to 90° perpendicular to the stress direction, where the crack exhibited a tearing state and rapidly expanded under the tensile stress, which was the main cause of sample fracture.

In the direction of 30~40° with the stress direction, the direction had two characteristics at the same time: one is that the direction had a large shear stress, and the stress was along the crack initiation direction. The second is that the angle between the direction and the direction of the vertical stress was about 60°. As discussed earlier, the primary crack causing the fracture of the specimen was approximately perpendicular to the stress direction, while the intragranular crack preferentially propagated along the <111> direction. If the direction of the main crack was assumed to be along the <111> direction of a certain grain, for a crack initiated in the crystal, an angle of 60° to the direction of the fracture means that the direction was very close to the other <111> directions. It is also easy to start a slip system through which dislocation slips and dislocation moved in the opposite direction. Therefore, the above-mentioned geometric rule of crack initiation was determined by the crystal structure of the material.

From the start of the crack initiation at 1535 cycles in Fig.3(b), to the 2413 cycles shown in Fig.3(c), the crack expanded but the expansion was quite slow, and then to 3078 cycles shown in Fig.3(d), cracks began to appear at the crack crossing grain boundaries, followed by rapid crack propagation, to 3123 cycles of sample fracture.

From the process of crack propagation, when the crack initiated in the crystal, the crack propagated directly through the crystal, and the expansion process was shown in Fig.3(d). The crack was actually
interconnected by two sets of cracks at an angle to each other to extend in the form of a broken line. This expansion should be related to its crystallographic orientation. However, no matter whether it is the macroscopic expansion direction of the crack or the actual expansion direction, the crack did not have a tendency to expand along the grain boundary. Even if the ultimate crack first propagated into a crack at the intersection of grain boundary, the direction of crack propagation was transgranular.

3.3. Fatigue at HT

High temperature in-situ fatigue test parameters could be found in Table 3. The sample thickness is 0.90 mm, the narrowest part of the width is 0.735 mm, the sample finally fractured at the cycle of 30879 cycles. The initiation and propagation of cracks during in-situ experiments could be found in Fig.6.

| Load/N  | Frequency/Hz | Stress Ratio | Load Stress/MPa | T/°C |
|---------|--------------|--------------|-----------------|------|
| 450     | 10           | 0.1          | 680             | 500  |

Fig.6 shows that crack initiation had not been observed on the surface of the sample when it was cycled to about 19988. At the cycle of 29166 cycles, crack initiation occurred on the surface of the sample, and cracks originated at the grain boundaries and propagated along the grain boundaries. At 29372 cycles, the crack propagated further, but the propagation direction was still generally along the grain boundary. By 29614 cycles, the crack continued to propagate into the interior of the sample. It should be noted that the crack did not propagate along the vertical direction of the stress, but propagated toward the intra-crystalline defect. By 30122 cycles, the crack transformed into transgranular propagation, and the propagation direction continued in a nearly straight line along the previous crack direction. By the 30359th cycle, the cracks showed a significant transgranular propagation, and the propagation direction remained in the original direction. The sample fractured when the stress was applied continuously to 30879 cycles.

Under room temperature fatigue test, when the fatigue stress was 80% of the yield strength, the alloy used in the test underwent about 11700 cycles of crack initiation and 12500 cycles of fracture, and only a few hundred cycles from crack initiation to sample fracture. At high temperatures, the same proportion of stress (80% of the yield strength of the sample) was maintained. The sample began to crack initiation when the cycle exceeded 29000 cycles. From crack initiation to sample fracture, it had undergone a cycle of about 2000 cycles. This was higher than room temperature experiments. This
should be due to recovery and recrystallization when the sample deformed at high temperatures. Ni is a low-level fault metal. Due to its wide width of extended dislocations, it is difficult to dynamically recover by cross-slip and edge dislocations, and the dynamic recovery rate is low, and the subdislocation density is large. Therefore, the tendency of dynamic recrystallization occurs.

Due to the slow crack propagation rate in the high temperature, it could track the crack length change in situ. Data in Fig.7 is measured by software and its projected length in the direction of main crack propagation was taken as the effective length of crack. The result is shown in Fig.7. It can be seen that at the initial stage of crack initiation, the crack grows along grain boundary at a relatively fast speed. The crack growth rate decreases as the crack turns to transgranular growth, but increases rapidly as the crack grows larger, indicating that the stress at the crack tip is highly concentrated, which is consistent with the performance of the crack in the normal temperature fatigue experiment.

Figure 7. Relationship between crack length and cycle time at 500°C

4. Conclusions

(1) Fracture characteristics of nickel-base superalloy at RT: when the cracks start, it mainly initiation along <111> direction of crystallographic orientation, the cracks are parallel, perpendicular, and at an angle f 53° and 60° to each other. Cracks initiated in the crystal propagate in a zigzag manner at an angle to each other, expansion direction and stress direction are vertical. The fatigue cycle required for crack initiation at the grain boundary is much lower than that in the crystal, and the crack propagation is much faster. At initial stage, cracks expand along the grain boundary to a certain extent and then propagate in a transgranular manner, the direction of which is approximately perpendicular to the stress direction.

(2) Fracture characteristics at high temperatures: the fatigue crack initiation time in alloys is much longer than room temperature, the cycle times required to initiate cracks on the premise of the same stress ratio exceed 15 times of room temperature. The time and length of fatigue crack propagation along the grain boundary are larger than those at room temperature, but the length of the transgranular expansion is slightly smaller.

5. References

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