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Abstract

The microstructural evolution of GH4169 alloy during long-term aging at 750 °C and its influence on low-cycle fatigue behavior were investigated. The results indicate that with increasing aging time, the size of the γ'' phase gradually increases while its volume fraction decreases; the size and volume fraction of the δ phase both increase; after long-term aging, the maximum cyclic stress response of the alloy under low-cycle fatigue decreases and the fatigue life is shortened. With increasing number of cycles, the cyclic stress response of the alloy sequentially exhibits characteristics of cyclic hardening, stabilization, and subsequent softening. During the aging process, the increased size and decreased volume fraction of the γ'' phase weaken its strengthening effect on the alloy, thereby reducing the stress response to cyclic strain; meanwhile, the formation of elongated needle-like δ phase and precipitate-free zones around it during aging provides a crack propagation path, resulting in a decrease in fatigue life.

Full Text

Influences of Long-Term Aging on Microstructure Evolution and Low Cycle Fatigue Behavior of GH4169 Alloy

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Abstract

This study investigates the microstructure evolution of GH4169 alloy during long-term aging at 750 °C and its influence on low-cycle fatigue behavior. The results show that with increasing aging time, the size of γ phases gradually increases while their volume fraction decreases; conversely, the size and volume fraction of δ phases increase. After long-term aging, the maximum cyclic stress response of the alloy decreases and fatigue life shortens. With increasing cycle number, the cyclic stress response of the alloy exhibits sequential characteristics of cyclic hardening, stability, and subsequent softening. The increase in γ phase size and decrease in volume fraction during aging weaken the strengthening effect, leading to reduced stress response under cyclic strain. Meanwhile, the formation of needle-like δ phases and surrounding precipitate-free zones during aging provides preferential paths for crack propagation, resulting in decreased fatigue life.

Keywords: GH4169 superalloy, long-term aging, microstructure evolution, low cycle fatigue

Introduction

Precipitation-strengthened nickel-based superalloys achieve superior high-temperature comprehensive properties primarily through solid solution strengthening and second-phase precipitation strengthening [1], making them suitable for high-strength applications involving creep-fatigue interaction at elevated temperatures [2]. GH4169 alloy, with its excellent thermal fatigue resistance, oxidation resistance, high-temperature strength, and creep performance, is widely used for manufacturing high-temperature structural components such as aero-engine turbine discs and industrial gas turbine discs [3-7].

However, due to its high degree of alloying and complex phase composition, combined with the long-term high-temperature service requirements of turbine discs [8], the microstructural stability of GH4169 alloy has attracted considerable attention [9,10]. Research [5] has shown that the temperature capability of modified GH4169 alloy can be improved to 680 °C. If applied to advanced gas turbine discs, the service temperature of modified GH4169 alloy is expected to reach 700 °C [11]. During actual service in high thrust-to-weight ratio aero-engines, short-term overtemperature and overload of the rotor are unavoidable to meet certain emergency load demands, which inevitably accelerates the microstructural evolution of turbine disc materials, thereby affecting their performance stability and service safety.

Consequently, as a turbine disc material, GH4169 alloy undergoes long-term exposure to high temperature and alternating loads, while mechanical constraints inevitably cause overload conditions. Overtemperature and overload lead to occasional high-strain low-cycle fatigue damage, significantly affecting service

safety and component lifespan [12,13]. Therefore, the low-cycle fatigue behavior of superalloys has received extensive attention. Parameters such as temperature, loading time, waveform, and strain amplitude are external factors influencing fatigue deformation behavior [14-20], while the alloy's inherent microstructural evolution characteristics play a decisive role [21].

Previous studies [22,23] have shown that during long-term high-temperature service, the primary strengthening phase γ in GH4169 alloy rapidly transforms into δ phase. The presence of an appropriate amount of short rod-like δ phase during fatigue deformation is beneficial for maintaining cyclic deformation resistance [2]; the size of γ phase decreases with repeated shearing by dislocations, and the deformation resistance consequently decreases [24]. Reviewing existing research, the focus has primarily concentrated on the relationship and mechanisms between service microstructural evolution and mechanical behavior of superalloys, while quantitative description and evaluation of the essential relationship between service microstructural evolution and mechanical property degradation or service life reduction have rarely been reported. As δ phase continues to grow, if it precipitates in a needle-like morphology at grain boundaries and within grains, it will strongly influence the fatigue deformation behavior and consequently affect alloy service life. Therefore, establishing a quantitative relationship between material service microstructural evolution and performance degradation or life reduction is important for effectively understanding the actual service damage degree of turbine disc components and reasonably evaluating their remaining safe service time, thereby providing a foundation for establishing an overall service life prediction model for turbine discs based on service microstructural evolution.

Accordingly, this study takes GH4169 alloy as the research object to investigate its microstructural evolution behavior and the influence rules and mechanisms on low-cycle fatigue behavior, quantitatively describing the correlation between microstructure and properties. This provides an effective basis for establishing quantitative relationships between microstructural evolution and performance degradation in typical superalloys, with the goal of providing a foundation for predicting fatigue deformation behavior based on material microstructural characteristics and establishing turbine disc life prediction models based on microstructural evolution.

1 Experimental Methods

The chemical composition of the GH4169 alloy used in this study is (mass fraction, %): C 0.023, Fe 17.30, Cr 17.92, Mo 2.98, Al 0.52, Ti 1.00, Nb 5.40, Mn 0.08, P 0.009, B 0.004, Ni balance. The alloy was melted by vacuum induction and vacuum consumable electrode melting. The ingot was homogenized and then hot-rolled into bars with a diameter of 100 mm, followed by hot forging into discs with a diameter of 230 mm and height of 95 mm. Square blanks

measuring $11\text{ mm} \times 11\text{ mm} \times 115\text{ mm}$ were taken from the center of the disc surface along the radial direction. The alloy underwent standard heat treatment (1050 °C for 1 h, air cooling; 720 °C for 8 h, furnace cooling at 50 °C/h to 620 °C, then 620 °C for 8 h, air cooling), designated as SHT. Samples after standard heat treatment were subjected to long-term aging at 750 °C for 500, 1000, 1500, and 2000 h. The aged alloys were machined into low-cycle fatigue specimens with a gauge section of 6 mm diameter and 30 mm length.

Low-cycle fatigue tests were conducted on an MTS-810 electro-servo hydraulic fatigue testing machine using fully reversed axial total strain-controlled tension-compression loading with a sinusoidal strain waveform. The total strain amplitude was $\Delta \epsilon/2 = \pm 0.5\%$, strain ratio $R_\epsilon = -1$, cyclic frequency $f = 0.3\text{ Hz}$, and tests were performed at room temperature. A GX71 inverted metallurgical microscope (OM) was used to observe the macrostructure, and quantitative metallographic analysis was employed to calculate the size and volume fraction of precipitates in the alloy. A JSM-6510 scanning electron microscope (SEM) was used to observe microstructure and fracture morphologies, and a JEOL-2100F transmission electron microscope (TEM) was used to characterize precipitate morphologies, dislocation configurations, and substructures.

2.1 Microstructural Evolution During Aging

Figure 1 [Figure 1: see original paper] shows OM images of GH4169 alloy after long-term aging at 750 °C. The grain sizes of the alloy after standard heat treatment and aging for 500, 1000, 1500, and 2000 h are 17.16, 16.48, 15.84, 16.32, and 15.02 μm , respectively. As shown in Figure 2 [Figure 2: see original paper], the spherical precipitates are γ phases, and with increasing aging time, the γ phase size increases and gradually forms disc-shaped γ phases. The disc-shaped γ phases continue to grow, and some transform into δ phases, resulting in a decrease in their volume fraction. Figure 3 [Figure 3: see original paper] illustrates the evolution process of increasing δ phase size and volume fraction. After aging for 500 h, δ phases nucleate at grain boundaries with granular and short rod-like distributions. By 1000 h, δ phases within grains grow into long needle-like shapes that traverse entire grains. Quantitative analysis results of precipitate size and volume fraction are shown in Table 1.

Research [29–31] has shown that the coherent strain between γ phases and the γ matrix is the main driving force for the transformation of γ phases to δ phases. With increasing aging time, the γ phase size increases, enhancing the coherent strain with the γ matrix, leading to misalignment on the close-packed planes of γ phases and the formation of stacking faults that develop into δ phases. The δ phases nucleated on stacking faults grow into the matrix and connect with each other, exhibiting a needle-like distribution. Needle-like δ phases generate γ -precipitate-free zones around them [32], as shown in Figure 3. It is evident that the width of the precipitate-free zones increases with aging time. Due to the

transformation of γ phases to δ phases, Nb atoms enrich in δ phases, while the matrix near δ phases becomes depleted in solute Nb atoms (Table 2), forming a microzone with high plasticity and low strength [23,33,34].

2.2 Low-Cycle Fatigue Strain-Stress Characteristics

The cyclic stress response curves of GH4169 alloy after different aging times at 750 °C are shown in Figure 4 [Figure 4: see original paper]. It can be seen that under a constant total strain amplitude, the cyclic stress response of the alloy exhibits characteristics of cyclic hardening, cyclic stability, and subsequent cyclic softening with increasing cycle number. With increasing aging time, the maximum cyclic stress response of the alloy decreases, and the low-cycle fatigue life reduces, as shown in Table 3.

3.1 Low-Cycle Fatigue Cyclic Stress Response Characteristics

During the initial stage of cyclic deformation, GH4169 alloy exhibits cyclic hardening. Research [24] has found that dislocations primarily shear fine γ phases during this stage, causing a strengthening effect. Figure 5 [Figure 5: see original paper] shows the dislocation configurations in GH4169 alloy aged at 750 °C for 1500 h. As shown in Figure 5b, after 50 loading cycles, dislocation networks form around δ phases in the alloy. It is evident that during the initial cyclic deformation stage, strong interaction occurs between δ phases and dislocations, hindering dislocation motion. Dislocations pile up around and between δ phases, increasing motion resistance and deformation resistance, which manifests macroscopically as cyclic hardening characteristics. Compared with the standard heat treatment condition, the alloy after long-term aging requires more loading cycles for its stress response to reach the maximum value. In the standard heat-treated alloy, no δ phases precipitate, and dislocations shearing γ phases cause cyclic hardening. Without further hindrance from δ phases to dislocation motion, the alloy quickly exhibits cyclic softening. The interaction between δ phases and dislocations under cyclic loading is the main cause of cyclic hardening in the alloy.

However, after dozens of loading cycles, dislocation generation and annihilation in the alloy reach equilibrium, and strengthening and weakening effects offset each other, macroscopically manifesting as saturated cyclic stress response. Figure 6 [Figure 6: see original paper] shows the stable cyclic stress-strain response curves of GH4169 alloy after different aging times at 750 °C. In this study, the total strain amplitude was $\pm 0.5\%$, corresponding to relatively low plastic strain and relatively small plastic deformation influence, so the alloy exhibited cyclic stability characteristics.

Under reciprocating cyclic loading, dislocations continuously shear fine spheri-

cal γ phases and disc-shaped γ phases while bypassing larger γ and γ phases, causing alloy strength degradation. With increasing cycle number, plastic deformation intensifies, and dislocations continuously move and pile up. Dislocation pile-up around δ phases, precipitate-free zones, and grain boundaries causes local plastic deformation incompatibility, leading to stress concentration that induces microcrack formation. As cycle number further increases, the number of microcracks caused by incompatible deformation increases, reducing the actual effective bearing area of the specimen. Consequently, the macroscopic cyclic stress response of the alloy decreases, softening intensifies until final fracture occurs.

Comparing the cyclic stress response curves of the alloy at different aging times in Figure 4 reveals that the peak cyclic stress response decreases with increasing aging time (Table 3). This is because during aging, γ phases in GH4169 alloy gradually grow and coarsen, with some transforming into δ phases, resulting in decreasing volume fraction of γ phases. Therefore, during cyclic deformation, dislocation motion in the alloy after short-term aging primarily involves shearing fine γ phases, while after long-term aging it mainly involves bypassing γ phases. With increasing aging time, the increasing size and decreasing volume fraction of γ phases weaken the strengthening effect, consistent with Orowan strengthening theory [35]. On the other hand, some γ phases transform into δ phases. With increasing aging time, δ phase size and volume fraction increase. Simultaneously, the volume fraction and width of γ -precipitate-free zones around δ phases increase, leading to decreased local deformation resistance. The combined effects of decreasing γ phase volume fraction, increasing δ phase volume fraction, growth of both phases, and increasing γ -precipitate-free zones around δ phases cause the peak cyclic stress response of the alloy to decrease with aging time.

3.2 Influence of δ Phase on Low-Cycle Fatigue Deformation Behavior

Figure 7 [Figure 7: see original paper] shows the morphologies of crack initiation zones in GH4169 alloy after low-cycle fatigue. As seen, fatigue cracks initiate near the specimen surface. In the stable crack propagation stage, numerous fatigue striations are observed, as shown in Figure 8 [Figure 8: see original paper]. Under high cyclic stress, the initial precipitate-free zones possess certain coordinated plastic deformation capability. With increasing cycle number, dislocation pile-up and stress concentration around δ phases further intensify. Figure 9 [Figure 9: see original paper] shows the morphology near the low-cycle fatigue fracture of GH4169 alloy aged at 750 °C for 2000 h. It can be seen that cracks easily propagate along needle-like δ phases or precipitate-free zones, further demonstrating that needle-like δ phases or precipitate-free zones around δ phases provide preferential paths for crack propagation.

During the rapid crack propagation stage, the fracture surface of the alloy aged

for 500 h shows numerous dimples in the final fracture zone, while the fracture surfaces of alloys aged for 1500 and 2000 h consist mainly of elongated features, as shown in Figure 10 [Figure 10: see original paper]. After aging for 500 h, the alloy contains a small amount of granular and short rod-like δ phases, with numerous disc-shaped γ phases precipitated in the matrix. Therefore, under cyclic stress, dimples easily form around γ phases and granular/short rod-like δ phases (Figure 10a [Figure 10: see original paper]). With increasing aging time, some γ phases in the alloy transform into long needle-like δ phases. Under cyclic loading, cracks tend to propagate along long needle-like δ phases and precipitate-free zones, forming elongated characteristic morphologies (Figures 10b and 10c). With increasing aging time, the proportion of elongated features in the rapid fracture zone of the fatigue fracture increases, mainly due to increased δ phase volume fraction and more precipitate-free zones in the alloy.

3.3 Low-Cycle Fatigue Life

As shown in Table 3, the fatigue life of GH4169 alloy decreases with increasing aging time. During long-term aging, the primary strengthening phase γ coarsens and partially transforms into δ phase, reducing its volume fraction. Meanwhile, δ phase size and volume fraction increase, and the auxiliary strengthening phase γ shows a coarsening trend [35]. These combined factors lead to decreased fatigue life with aging time.

As seen in Figure 6, the low-cycle fatigue plastic strain amplitude Δp slightly increases with aging time. As previously described, during long-term aging, on one hand, γ phase size increases and volume fraction decreases, increasing interparticle spacing; on the other hand, the width and volume fraction of γ -precipitate-free zones around δ phases increase. Both factors improve alloy plastic deformation capability and increase Δp during cyclic deformation. Typically, larger Δp corresponds to higher low-cycle fatigue life. However, this study found that the low-cycle fatigue life of GH4169 alloy shows a decreasing trend with aging time. Tensile test results after long-term aging show that alloy elongation decreases with aging time [36], with the unidirectional tensile elongation after aging at 750 °C for 2000 h decreasing from approximately 20% in the SHT condition to below 10%. Analysis indicates that the decreased elongation originates from the precipitation of numerous needle-like δ phases after long-term aging. When the Schmid component of the applied load in each grain is perpendicular to needle-like δ phases, dislocation pile-up creates stress concentration, easily causing bending and fragmentation of needle-like δ phases (Figure 11 [Figure 11: see original paper]); when the load component is parallel to needle-like δ phases, cracks easily propagate along needle-like δ phases or precipitate-free zones (Figure 9). In other words, after long-term aging, the increased size of δ phases, particularly the increased amount of needle-like δ phases, causes premature cracking during deformation, and cracks easily propagate along δ phases, reducing the low-cycle fatigue life of the alloy.

Based on the above analysis, during long-term service under cyclic loading, GH4169 alloy exhibits reduced low-cycle fatigue life due to microstructural evolution from long-term aging. The microstructural evolution characteristics under near-service conditions were obtained, such as in this study where the γ phase volume fraction decreased to 18% while the δ phase volume fraction increased to 19.4% after aging. Since the amount of γ phase is much less than that of δ phase, its separate discussion is omitted here. The quantitative changes in low-cycle fatigue resistance and life can be inferred: after aging for 500 h, compared with the standard heat-treated condition, the maximum cyclic stress decreases by 8.3% and fatigue life decreases by 14.8%; after aging for 2000 h, compared with the standard heat-treated condition, the maximum cyclic stress decreases by 32.4% and low-cycle fatigue life decreases by 35%.

Conclusions

1. During long-term aging of GH4169 alloy at 750 °C, γ phases coarsen with increasing aging time. γ phases increase in size and decrease in volume fraction, partially transforming into δ phases. δ phases evolve from granular to short rod-like and then to needle-like morphologies, with increasing size and volume fraction, generating γ -precipitate-free zones around them that increase in both volume fraction and width.
2. After long-term aging at 750 °C, GH4169 alloy exhibits cyclic hardening, cyclic stability, and cyclic softening characteristics during low-cycle fatigue deformation. However, with increasing aging time, both the maximum cyclic stress response and low-cycle fatigue life of the alloy decrease. During long-term aging, γ phases coarsen, γ phases increase in size and decrease in volume fraction, weakening the strengthening effect. The transformation of numerous γ phases into δ phases reduces alloy strength, while cracks easily propagate along coarsened needle-like δ phases and their surrounding precipitate-free zones.
3. The performance degradation of GH4169 alloy is mainly caused by the growth, coarsening, and transformation of γ phases into δ phases. When the γ phase volume fraction decreases to 18% and the δ phase volume fraction reaches 19.4%, the maximum cyclic stress decreases by 8.3% and low-cycle fatigue life decreases by 14.8%; when the δ phase volume fraction reaches 19.4%, the maximum cyclic stress decreases by 32.4% and low-cycle fatigue life decreases by 35%.

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