Research Progress on Microstructural Stability and Mechanical Behavior of Advanced Nickel-Based Single Crystal Superalloys: Postprint

Authors: Jin Tao, Zhou Yizhou, Wang Xinguang, Liu Jinlai, Sun Xiaofeng, Hu Zhuangqi

Date: 2023-03-19T00:00:00+00:00

Abstract

From the two aspects of microstructural stability and mechanical behavior of advanced nickel-based single crystal superalloys, this article briefly reviews the main research progress on γ' phase rafting, TCP phase precipitation, high-temperature and ultra-high-temperature low-stress creep, as well as low-cycle and thermomechanical fatigue. The addition of the alloying element Ru enhances the high-temperature low-stress creep life of the alloy, but also indirectly promotes the occurrence of the topological inversion phenomenon. With increasing aging time and aging temperature, the refractory element content in the phase increases significantly; with increasing applied stress, the precipitation amount of phase increases, whereas the opposite occurs under compressive stress. During precipitation, the phase forms numerous planar defects, which facilitate the nucleation of other TCP phases such as the P phase and R phase. During high-temperature low-stress creep, another important type of a<010> superdislocations emerges in nickel-based single crystal superalloys, moving slowly within the γ' phase via a combination of glide and climb. Under ultra-high-temperature creep conditions, an incubation period for creep acceleration appears, which is associated with varying degrees of widening of the γ matrix at ultra-high temperatures. The addition of Ru significantly reduces the stacking fault energy of the alloy, which during low-cycle fatigue can induce complex deformation mechanisms such as stacking faults penetrating γ/γ' interfaces and a/6<112> Shockley trailing dislocations shearing into the γ' phase. During thermomechanical fatigue, the location of crack initiation, microstructural evolution, and oxidation resistance all influence the life of nickel-based single crystal superalloys.

Full Text

Preamble

ACTA METALLURGICA SINICA, Vol. 51, No. 10, October 2015, pp. 1153-1162

Research Progress on Microstructural Stability and Mechanical Behavior of Advanced Ni-Based Single Crystal Superalloys*

JIN Tao, ZHOU Yizhou, WANG Xinguang, LIU Jinlai, SUN Xiaofeng, HU Zhuangqi (Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016)

Abstract: Ni-based single crystal superalloys have been widely used to produce turbine blades for advanced aero-engines because of their excellent high temperature microstructural stability and comprehensive mechanical properties. However, due to effects of high temperature and complex stresses in service, the microstructures of superalloys might gradually evolve and fail in different modes. The present paper reviews progress on microstructural stability and mechanical behavior including γ' phase rafting, TCP phase precipitation, high temperature creep, low cycle fatigue and thermomechanical fatigue of single crystal superalloys. The addition of Ru improves the creep life of superalloys, but also indirectly promotes the occurrence of "topological inversion". On the other hand, with increasing aging temperature and time, the contents of refractory elements in the μ phase rise significantly. With increasing applied tensile stress, more μ phase precipitates from the γ matrix, whereas the inverse tendency occurs under compressive stress. Numerous planar defects are formed during precipitation of the μ phase, and these defects promote nucleation of P and R phases. During high temperature and low stress creep, an important dislocation $a\langle 010\rangle$ superdislocation is observed, which moves in the γ' phase slowly by a combination of glide and climb. Under very high temperature, incubation with accelerating creep rate occurs before the primary stage, which relates to the widening process of the γ channels. Finally, the stacking fault energy is significantly reduced after Ru additions, and thus a series of complex deformation mechanisms occur during low cycle fatigue, e.g. stacking faults penetrating γ/γ' interface, trailing $\frac{a}{6}\langle 112\rangle$ Shockley dislocations shearing into the γ' phase. During thermomechanical fatigue, the life of superalloys is influenced by the site of crack initiation, microstructural evolution and oxidation resistance.

KEY WORDS Ni-based single crystal superalloy, microstructural stability, mechanical behavior

Ni-based single crystal superalloys are widely used in aerospace and other fields due to their high room-temperature and high-temperature strength, good ductility and fracture toughness, excellent creep and fatigue resistance, good high-temperature microstructural stability and service reliability, and good oxida-

tion and corrosion resistance [1~3]. Extensive research has been conducted on Ni-based single crystal superalloys both domestically and internationally, with Chinese research institutions such as the Institute of Metal Research, Chinese Academy of Sciences and Beijing Institute of Aeronautical Materials carrying out fruitful work in recent years. To date, based on temperature capability, Ni-based single crystal superalloys can be basically divided into five generations, as shown in Figure 1 [Figure 1: see original paper][1]. The addition of large amounts of alloying elements (such as Re, Cr, Mo, Ta, and W, etc.) is the main reason why Ni-based single crystal superalloys have relatively high temperature capability [2,3]. In particular, the addition of Re significantly improves the high-temperature creep performance of alloys [1~3]. However, due to the addition of large amounts of Re and other refractory elements, the alloy system becomes unstable, and topologically close-packed (TCP) phases [4~7] are prone to precipitate during service, thereby limiting further improvement of high-temperature properties of single crystal superalloys. To solve this problem, recent studies [8,9] have made full use of the role of Re and other refractory elements in superalloys, improved microstructural stability by adding platinum group elements such as Ru, and further enhanced comprehensive mechanical properties, developing fourth- and fifth-generation Ni-based single crystal superalloys with better microstructural stability and longer creep life.

In the development of single crystal superalloys, good high-temperature microstructural stability and comprehensive mechanical properties have always been the goals pursued by researchers, but the addition of large amounts of refractory elements often makes it difficult to achieve both. This work focuses on the high-temperature microstructural stability and mechanical behavior of Ni-based single crystal superalloys, briefly introducing relevant research progress on advanced Ni-based single crystal superalloys.

1.1 Microstructure of Single Crystal Superalloys

Although Ni-based single crystal superalloys contain numerous alloying elements with very complex interactions, their microstructure is relatively simple, consisting mainly of the γ matrix phase and γ' precipitate phase (Ni_3Al), as shown in Figure 2 [Figure 2: see original paper][10,11]. The γ phase is an fcc disordered solid solution based on Ni; the γ' phase is an ordered fcc precipitate with an L12 superlattice structure and is the main strengthening phase of Ni-based single crystal superalloys. The γ' phase is embedded in the γ matrix in a relatively regular manner and maintains a coherent relationship with it ($(100)_{\gamma}$ $(100)_{\gamma'}$, $[001]_{\gamma}$ $[001]_{\gamma'}$).

The size, morphology, and volume fraction of constituent phases significantly affect the mechanical properties of alloys. For most Ni-based single crystal superalloys, the γ' phase is cubic with a size of 0.2–0.5 μm and a volume fraction of about 65% [12]. The lattice constant of the γ' phase is slightly smaller than that of the γ phase, with a difference on the order of 10^{-3} [13], resulting in misfit stress (dislocations) at the γ/γ' interface (Figure 2b). The width of γ

phase channels is between 50–100 nm (depending on alloy composition and heat treatment regime), and the wider the γ phase channel, the easier for dislocations to move. As mentioned above, Ni-based single crystal superalloys contain numerous alloying elements and have misfit stress between γ/γ' phases, leading to microstructural instability under high temperature and complex stress, with phenomena such as γ' phase rafting and TCP phase precipitation and growth, which directly affect the mechanical behavior of alloys.

1.2 Rafting of γ' Phase

Generally, under long-term high-temperature aging or high-temperature low-stress creep, the rafting process in single crystal superalloys is unavoidable, and almost all Ni-based single crystal superalloys will undergo rafting. Studies [14] have shown that the elastic stress field between γ/γ' phases determines the rafting direction of the alloy: when the misfit between γ/γ' phases is negative, γ' phase undergoes N-type rafting (rafting direction perpendicular to the applied stress direction) under tensile conditions and P-type rafting (rafting direction parallel to the applied stress direction) under compressive conditions; when the misfit between γ/γ' phases is positive, the situation is reversed. In fact, the misfit of the alloy changes with temperature, so the rafting type occurring during high-temperature creep depends on the sign of misfit at that temperature and the direction of applied stress.

Under high-temperature low-stress creep conditions, the γ' phase rafted structure is basically formed at the beginning of steady-state creep [15]. During subsequent creep, the rafted structure continues to evolve, with the length of rafted γ' phase continuously increasing and the γ matrix being continuously "cut" by dislocations. Under certain creep temperatures and stresses, the so-called topological inversion phenomenon [16,17] may occur. Topological inversion is the result of long-term rafting in Ni-based single crystal superalloys and has a significant effect on creep life. Ru addition improves the high-temperature low-stress creep life of alloys but also indirectly promotes the occurrence of topological inversion [17]. Creep experiments on high-temperature alloys with different Ru contents under ultra-high temperature conditions also show that Ru addition tends to cause topological inversion [16].

Theoretically, the closer the misfit is to zero, the more stable the two-phase structure and the slower the rafting process. However, the misfit of the alloy changes with temperature, so in actual alloy design, efforts should be made to make the high-temperature misfit close to zero. In addition, the rafted two-phase structure can hinder the movement of matrix dislocations, enabling the alloy to maintain high strength at high temperatures. Therefore, the rafted structure is beneficial for maintaining a longer steady-state creep stage [17]. Future research needs to further study the γ/γ' interface at the atomic scale and through computational simulation, clarify the evolution behavior of the interface, and explore how to slow down the rafting process to improve microstructural stability.

1.3 Topologically Close-Packed Phases in Single Crystal Superalloys

Due to the presence of large amounts of refractory elements, Ni-based single crystal superalloys will precipitate some refractory element-rich intermetallic phases with complex crystal structures during long-term thermal exposure or service. These phases contain only tetrahedral interstices and are highly close-packed. Because of this special close-packed structure, these phases are called TCP phases [18,19]. TCP phases in Ni-based single crystal alloys include σ, μ, P, and R phases, with their crystallographic information listed in Table 1 [18,19].

From a thermodynamic perspective, the nucleation driving force for TCP phases is controlled by the supersaturation of TCP phase-forming elements in the γ phase. Studies [20~23] suggest that the "reverse partitioning" of alloying elements caused by Ru addition, the increased solubility of elements such as Re and W in the γ matrix, and the change in lattice misfit between the γ matrix and TCP phases may be the main reasons for suppressing TCP phase nucleation. TCP phase growth is controlled by element diffusion [20], and Ru addition can inhibit TCP phase precipitation. However, so far, there is no direct evidence that Ru or other element additions can significantly reduce the diffusion rate of TCP phase-forming elements. What is certain is that Ru addition changes the interactions between alloying elements, thereby affecting the growth process of TCP phases. Recent TEM in-situ observation studies of TCP phase precipitation at high temperatures [24] show that Ru addition can simultaneously reduce both nucleation and growth rates of TCP phases. Although high temperature reduces TCP phase nucleation rate, it increases its growth rate, ultimately leading to accelerated TCP phase precipitation. In summary, although many researchers have attempted to explain the nucleation and growth processes of TCP phases and the mechanism by which Ru suppresses TCP phase formation, no unified view has been reached.

The morphology and amount of TCP phases significantly affect the mechanical properties of alloys. Currently, more research has been conducted on the μ phase. In Re-containing alloys, the μ phase usually appears as rod-like, plate-like, and particle-like shapes. Large plate-like μ phases destroy the continuity of γ/γ' two-phase structure, leading to non-uniform deformation during alloy creep. A small amount of rod-like μ phases has little effect on alloy performance but "softens"the γ matrix. Although large particle-like μ phases can cause stress concentration, it is insufficient to initiate cracks. Long-term aging time, temperature, and alloying elements all affect the composition and amount of μ phase. In the later stage of long-term aging compared to the early stage, and at high-temperature aging compared to low-temperature aging, the refractory element content in μ phase increases significantly. Increased Re content reduces alloy stability, and the amount of TCP phases precipitated during high-temperature aging increases significantly, but does not change the type of TCP phases (all are μ phase). During high-temperature creep, the magnitude and direction of applied

stress, misfit stress between γ/γ' phases, and evolution of γ' phase can all affect μ phase precipitation. As applied tensile stress increases, the amount of μ phase precipitation increases, while the opposite is true under compressive stress, mainly because applied stress changes the interface misfit relationship between γ and μ phases [25].

The microstructure of TCP phases is relatively complex, with various defects. Studies on TCP phase precipitation behavior in high Re single crystal superalloys with different Ru contents after isothermal aging at 1100 ℃ for 1000 h and creep at 1100 ℃, 150 MPa show that the precipitated TCP phases are all μ phases containing large amounts of planar defects, including twins, micro-twins, stacking faults, etc. (Figure 3 [Figure 3: see original paper][26]). Ru addition can inhibit TCP phase precipitation but simultaneously promotes an increase in the number of internal defects in μ phase. In addition, the precipitation process of μ phase is very complex, often coexisting with other types of TCP phases (P or R phases, etc.) (Figure 4 [Figure 4: see original paper][26]). P or R phases nucleate at stacking fault positions in μ phase and continuously consume μ phase to grow, but the matrix remains μ phase, with P and R phases accounting for a small proportion. Further research on the effect of Ru on TCP phase precipitation behavior in Re-free single crystal superalloys [27] shows that during long-term aging at 980 ℃, because Ru reduces the coherent interface energy ΔG_{LD} between TCP phase and γ phase, decreases the resistance to TCP phase nucleation, and promotes W segregation in dendrite arms, it leads to increased TCP phase precipitation. However, during long-term aging at 1100 ℃, the amount of TCP phases first increases and then decreases. Moreover, Ru addition shifts the alloy' s TCP phase temperature-time-transformation curve to the left.

In addition, the σ phase is relatively common under certain temperature conditions, while literature on P and R phase precipitation is limited, especially R phase which is rarely seen among TCP phases precipitated in Ni-based single crystal alloys [23]. Some studies [28] have also found that Ru addition promotes TCP phase precipitation. This introduces controversy about the essential role of Ru in superalloys, requiring more detailed work to clarify.

It is generally believed that large amounts of plate-like TCP phases cause serious damage to alloy creep properties. Therefore, avoiding TCP phase precipitation is an important aspect of alloy design. TCP phase formation is mainly controlled by electronic factors and is related to the alloy' s electron vacancy concentration. The precipitation of TCP phases can be predicted by calculating the electron vacancy number N_V in the alloy [2].

2.1 High-Temperature Low-Stress Creep Behavior

Although hollow turbine blades have been designed with relatively ideal complex cooling channels, the surface temperature at the blade edge is still very high during actual service, while also being subjected to centrifugal stress. Therefore,

high-temperature creep damage caused by the combined action of temperature and stress is an important failure mechanism for Ni-based single crystal superalloys. Based on changes in creep rate, the creep process can generally be divided into three stages: primary creep stage, steady-state creep stage, and tertiary creep stage.

In the primary creep stage, the most obvious characteristic is a relatively high creep rate. Microscopically, a large number of $\frac{a}{2}\langle 101\rangle\{111\}$ dislocations on different slip systems are activated. Leading screw dislocation segments undergo cross-slip on different $\{111\}$ planes in horizontal matrix channels, leaving 60° mixed dislocations at γ/γ' interfaces [11,29]. Since there are few dislocations in the γ matrix at the beginning of creep deformation, a large number of dislocations can nucleate and multiply rapidly in the matrix, which manifests as a high creep rate on the macroscopic creep curve, with rapid accumulation of plastic strain. As creep proceeds, dislocations begin to pile up at γ/γ' interfaces, and dislocation movement gradually becomes difficult, which manifests as a rapid decrease in strain rate on the creep curve. Interface dislocations of different slip systems in the matrix begin to react under the combined effects of temperature, applied stress, misfit stress, and mutual stress fields between dislocations, gradually evolving into interface dislocation networks [30].

A significant decrease in creep rate marks the entry into the steady-state creep stage. As creep continues, the interface dislocation network further evolves, gradually forming more regular hexagonal or quadrilateral structures [30]. Most researchers believe that the density of interface dislocation networks is related to the misfit at the γ/γ' interface. The larger the misfit, the denser the dislocation network, because dense interface dislocation networks can effectively release misfit stress. Zhang et al. [31] first proposed that dense dislocation networks can effectively hinder matrix dislocations from shearing into the γ' phase, extending the steady-state creep stage. However, forming dense dislocation networks requires the alloy to have a more negative (positive) misfit, and the larger the absolute value of misfit, the faster the alloy rafting process. Recently, studies on the coupling relationship between Re segregation and interface dislocation networks during high-temperature low-stress creep of Ni-based single crystal superalloys [32] found that there are three different types of protrusions at the γ/γ' interface, and these protrusions are related to the segregation of Re, Cr, and Co. This research helps understand the relationship between interface dislocations and elements.

In the steady-state creep stage, as creep proceeds, a small number of different types of superdislocations begin to shear into rafted γ' phase. There are mainly two types of superdislocations: $a\langle 101\rangle$ superdislocation and $a\langle 010\rangle$ superdislocation. Among them, the $a\langle 101\rangle$ superdislocation is the most common and important dislocation type in Ni-based single crystal superalloys under various deformation conditions (tensile, medium-temperature high-stress and high-temperature low-stress creep, low-cycle fatigue, and thermomechanical fatigue, etc.). It is generally believed that this dislocation is formed by the combination

of two $\frac{a}{2}\langle 101\rangle$ dislocations with the same Burgers vector on the same {111} plane at the γ/γ' interface [33]. When shearing into the γ' phase, the spacing between the two $\frac{a}{2}\langle 101\rangle$ partial dislocations is determined by the antiphase boundary (APB) energy in the γ' phase. Generally, because the APB energy of the γ' phase is very high, the spacing between the two $\frac{a}{2}\langle 101\rangle$ partial dislocations is very small. During steady-state creep, due to the hindering effect of interface dislocation networks and high APB energy, the number of $a\langle 101\rangle$ superdislocations shearing into the γ' phase is very small compared with matrix dislocations. Therefore, plastic strain accumulation in the steady-state creep stage is not significant.

During high-temperature low-stress creep, another important dislocation in Ni-based single crystal superalloys is the $a\langle 010\rangle$ superdislocation. Studies on high-temperature low-stress creep behavior of two Ni-based single crystal superalloys with different Ru contents [17,34] found that $a\langle 010\rangle$ superdislocations could be observed under all creep conditions involved in the study, and they were divided into two categories: long straight superdislocations and zigzag-shaped superdislocations. It was also found that under high-stress conditions at 1000 ℃, the steady-state creep rate was lower than under low-stress conditions at 1100 and 1140 ℃, which may be related to the movement mode of $a\langle 010\rangle$ superdislocations. Some studies [35,36] believe that $a\langle 010\rangle$ superdislocations are formed by the reaction of two $\frac{a}{2}\langle 011\rangle$ matrix dislocations with different Burgers vectors meeting at the γ/γ' interface. After shearing into the γ' phase, they lie on {001} planes. Their dislocation cores are not compact, consisting of two partial dislocations with different Burgers vectors. They move slowly in the γ' phase through a combination of glide and climb, with climb controlling the movement rate of superdislocations. Therefore, the movement rate of superdislocations is higher at high temperatures.

Unlike $a\langle 101\rangle$ superdislocations, $a\langle 010\rangle$ superdislocations have their own morphological characteristics. By comparing the morphologies of the two types of superdislocations [34], it was found that dislocation lines of $a\langle 101\rangle$ superdislocations are mainly along [100] and [010] directions, while dislocation lines of $a\langle 010\rangle$ superdislocations are mainly along [110] and $[\bar{1}10]$ directions (both projected onto the (001) plane), which may be related to the energy states of the two types of dislocations. In addition, it was found that at the same temperature, the stress required for $a\langle 101\rangle$ superdislocations to shear into the γ' phase is somewhat higher than that for $a\langle 010\rangle$ superdislocations. Overall, research on the role of $a\langle 101\rangle$ superdislocations in high-temperature creep processes is still insufficient, particularly regarding their formation conditions, movement modes, and role in creep.

The tertiary creep stage is characterized by a sharp increase in creep strain, which is a strain-softening stage, also known as the creep acceleration stage. In Ni-based single crystal superalloys, the two most important causes for the significant increase in creep rate during the tertiary stage are the increase in mobile dislocation density and the occurrence of topological inversion [26]. In

this stage, creep deformation is mainly achieved by dislocations cutting the γ' phase, including $a\langle 101\rangle$ superdislocations, 60° or screw-type superdislocations, $\langle 010\rangle$ superdislocations, and even stacking faults [37]. In the tertiary creep stage, the interface dislocation network becomes unstable, with dislocations obviously bowing out and moving. In addition, casting shrinkage pores at interdendritic regions have a significant effect on the final fracture of alloys, because microcracks causing fatal damage to the alloy mostly initiate at pores and propagate rapidly during the tertiary creep stage [38]. Furthermore, phenomena such as corrosion, oxidation, micropore aggregation, and crack extension occurring in the final creep stage are also important factors in alloy damage. In addition to the above creep mechanisms, the relationship between the disappearing γ matrix and dislocations during rafting in high-temperature low-stress creep and its effect on creep rate have been studied, and complex dislocations and dislocation arrays have been observed in γ' precipitates [15], indicating that high-temperature low-stress creep still requires further investigation.

2.2 Ultra-High Temperature Creep Behavior

Currently, advanced Ni-based single crystal superalloys can be used for long-term service at temperatures up to 1100 ℃ with certain strength and microstructural stability. For such alloys, temperatures exceeding 1150 ℃ can be called ultra-high temperatures. Under some emergency conditions (such as when one engine fails in a twin-engine helicopter), the short-term service temperature of the alloy can be as high as 1200 ℃ [39]. Therefore, research on the creep behavior of alloys under extreme conditions such as ultra-high temperatures is particularly important. So far, there have not been many studies on ultra-high temperature creep of Ni-based single crystal superalloys.

Studies on ultra-high temperature creep behavior of three advanced Ni-based single crystal superalloys with different Ru contents under different temperatures and stresses [26] found that under conditions of 1150 ℃, 100 MPa and 1180 ℃, 70 MPa, the ultra-high temperature creep curves of the three alloys all exhibited four different creep stages: incubation period, primary creep stage, secondary creep stage, and tertiary creep stage. The creep rate showed a trend of first increasing, then decreasing, and then increasing again. It is believed that the varying degrees of widening of γ matrix channels at ultra-high temperatures are the reason for the very short incubation period appearing before the primary creep stage, and the length of the incubation period depends on the coarsening of γ' phase.

Under the above two conditions, when creep strains reach (1.0$±0.2)±$0.05)% respectively, creep enters the acceleration stage, the creep rate increases sharply, and fracture occurs within the subsequent tens of hours [26]. This research result is consistent with the viewpoint in literature [38]. Because Ru addition significantly reduces the minimum creep rate and extends the steady-state creep stage, the ultra-high temperature low-stress creep life of alloys significantly improves with Ru addition. Overall, Ru addition can reduce interface dislocation network

spacing, improve the stability of interface dislocation networks, make interface dislocation network distribution more uniform, and simultaneously improve the high-temperature stability of γ' phase and its volume fraction at ultra-high temperatures. These factors are the reasons for reducing the steady-state creep rate, extending the duration of the second creep stage, and ultimately improving alloy creep life. Studies on ultra-high temperature creep behavior of MC-NG series alloys [40] also believe that the high γ' phase solvus temperature of the alloy is the reason for its long ultra-high temperature creep life, meaning the alloy microstructure still contains a high volume fraction of γ' phase under ultra-high temperature conditions.

The above three alloys with different Ru contents did not exhibit topological inversion during ultra-high temperature creep deformation under the two conditions. The origin of the tertiary creep stage is due to unstable crack propagation at the edges of pores (including casting shrinkage pores and pores induced by topologically close-packed phases) in the necking region [26]. This conclusion is also consistent with the research results in literature [38]. In summary, under the effect of temperature, various thermally activated processes accelerate during ultra-high temperature creep. Therefore, under most ultra-high temperature conditions, the creep life of alloys decreases to varying degrees.

2.3 Low-Cycle Fatigue Behavior

Low-cycle fatigue (LCF) is an important damage mechanism for single crystal blades in gas turbine engines during service.

2.3.1 Low Temperature Range

Planar slip is the main characteristic of dislocation slip in Ni-based single crystal superalloys during room-temperature low-cycle fatigue deformation [41]. Slip bands can continuously shear γ and γ' phases, and their appearance is related to initial work hardening, which subsequently leads to cyclic softening [42]. During deformation at room temperature, the density of slip bands increases significantly with increasing total strain amplitude. Studies on low-cycle fatigue behavior of Ni-based single crystal superalloys with different Re contents in the low-temperature stage [41] show that from room temperature to 250 ℃, a large number of slip bands appear on the alloy surface. Slip bands can directly cut γ' phase, which has almost no strengthening effect, and cracks propagate along slip bands. This research result is consistent with the conclusion in literature [43]. However, studies on room-temperature low-cycle fatigue of Ru-containing Ni-based single crystal superalloys [44] found that after cycling for a certain number of cycles, the alloy did not show cyclic softening but showed obvious cyclic hardening behavior, and the number of slip bands in the sample was small. It was also found that a small number of dislocations had three-dimensional distribution characteristics, and this three-dimensionally distributed dislocation is an important component of slip bands after low-cycle fatigue at 900 ℃, indicating that dislocation movement is very complex during actual fatigue processes.

In addition to generating slip bands, the following deformation mechanisms can be observed during low-cycle fatigue in the low-temperature range [42~44]: (1) $a\langle 101\rangle$ superdislocations shearing γ' phase; (2) $a\langle 101\rangle$ superdislocations decomposing during the process of shearing γ' phase, generating stacking fault loops; (3) a small number of $\frac{a}{2}\langle 101\rangle$ matrix dislocations decomposing at the γ/γ' interface, with $\frac{a}{3}\langle 112\rangle$ partial dislocations shearing into precipitates and generating stacking faults; (4) in Ru-containing alloys, $\frac{a}{2}\langle 101\rangle$ dislocations decomposing in the matrix and generating stacking faults.

2.3.2 Medium and High Temperature Range

As deformation temperature increases, the cyclic stress response curves of Ni-based single crystal superalloys begin to show differences. Studies on medium- and high-temperature low-cycle fatigue behavior of DD98 Ni-based single crystal alloy [45] show that at 750 ℃, the alloy exhibits stable cyclic stress response behavior, with only short-term softening at the end of cycling, while at 950 ℃, the alloy shows obvious cyclic softening behavior. The fatigue life of the alloy decreases with increasing temperature, but the effect of temperature decreases at high strain amplitudes. With increasing total strain amplitude, the proportion of fatigue crack initiation and early propagation on the fracture surface decreases accordingly, and fatigue failure shows multi-source cracking. Research results in literature [46,47] are consistent with the above conclusions, and also found that the alloy shows obvious tension-compression asymmetry during medium-temperature fatigue. However, recent research [44] points out that the fourth-generation single crystal superalloy containing Ru shows slight work hardening during deformation at 760 ℃, obvious work hardening during deformation at 900 ℃, but basically no change in cyclic stress during high-temperature deformation at 980 ℃, with no obvious softening behavior, which may be related to the alloy system studied.

During medium- and high-temperature fatigue, the stacking fault energy of the alloy increases with temperature, leading to significant changes in microscopic deformation mechanisms. Studies on low-cycle fatigue behavior of Ni-based single crystal superalloys [41] found that slip bands are difficult to observe above the peak temperature T_p, and the dislocation slip mode gradually changes from planar slip to wavy slip. In the medium- and high-temperature range, dislocation movement modes mainly include slip, climb, dislocation interactions, formation of irregular dislocation networks at the γ/γ' interface, and a few stacking faults shearing γ' phase [42,44,47~49]. With increasing temperature, the number of slip bands decreases, but cracks still propagate along slip bands. At 900 ℃, alloy deformation is relatively uniform, but creep and oxidation intensify alloy fatigue damage [41]. Recent research results [44] show that after low-cycle fatigue from room temperature to 980 ℃ in Ru-containing alloys, stacking faults appear in both matrix and precipitates, indicating that Ru addition significantly reduces alloy stacking fault energy and changes alloy fatigue deformation mechanisms to some extent. After low-cycle fatigue at 900 ℃, evolving slip bands were ob-

served, with cross-slip dislocations, entangled dislocations, and stacking faults in both matrix and precipitates being the main components (Figure 5 [Figure 5: see original paper][44]).

In addition to the several deformation mechanisms mentioned above, when studying low-cycle fatigue of two Ni-based single crystal superalloys with different Ru contents, it was found that stacking faults can penetrate the γ/γ' interface, $\frac{a}{6}\langle 112\rangle$ Shockley trailing dislocations can shear into the γ' phase, and Lomer-Cottrell dislocations can be observed in the γ' superlattice structure [15]. These are non-negligible deformation mechanisms when analyzing the low-cycle fatigue process of Ni-based single crystal superalloys.

2.4 Thermomechanical Fatigue Behavior

Thermomechanical fatigue (TMF) refers to the low-cycle fatigue behavior of materials when they simultaneously experience temperature and load changes. Compared with isothermal low-cycle fatigue, thermomechanical fatigue can more realistically simulate the stress and deformation of materials during service, thus enabling more accurate life assessment of materials.

Studies on thermomechanical fatigue of SRR99 single crystal superalloy [50] show that during the high-temperature half-cycle of IP- (in-phase, i.e., maximum strain corresponds to highest temperature) and OP- (out-of-phase, i.e., maximum strain corresponds to lowest temperature) TMF, the alloy shows initial cyclic softening at all strain amplitudes, while showing initial cyclic hardening characteristics during the low-temperature half-cycle. Under small strain amplitudes of IP-TMF, TMF specimens fail by micropore aggregation, with cracks initiating on the specimen surface and propagating under the combined action of oxidation and creep. With increasing mechanical strain amplitude, fatigue caused by cyclic plastic deformation becomes the main reason for IP-TMF fracture. Under OP conditions, fatigue cracks mainly initiate on the specimen surface and propagate rapidly under tensile stress, with oxidation and creep effects being not obvious, thus it should be a stress-controlled fatigue process. Due to differences in crack initiation and propagation mechanisms, the IP-TMF life of the alloy is higher than OP-TMF. This research result is consistent with the thermomechanical fatigue behavior study results of CMSX-6 Ni-based single crystal superalloy [51]. However, studies on DD8 single crystal alloy IP-TMF and OP-TMF [52] found that IP-TMF life is lower than OP-TMF, and believed that differences in crack initiation locations cause the reversal of alloy life. Under IP-TMF conditions, cracks initiate at internal pores in the alloy, while under OP-TMF conditions, cracks initiate on the alloy surface. Although literature [50] believes that crack initiation locations in IP-TMF and OP-TMF are basically the same, the results are completely opposite, indicating that alloy fatigue fracture is not only related to crack initiation location but also to changes in internal microstructure and oxidation resistance of the alloy.

In recent years, new progress has been made in the fatigue fracture mechanism

of single crystal superalloys, with fatigue cracking caused by twins becoming a research hotspot. Studies [53,54] found that during thermomechanical fatigue of TMS-82 and CMSX-4 Ni-based single crystal superalloys, twins play a very important role in alloy deformation localization and fracture processes. However, twins were not observed after thermomechanical fatigue failure of AM3 [55], SRR99 [50], and DD98 [56] Ni-based single crystal superalloys, with alloy deformation mainly based on dislocation slip and stacking fault shearing of γ' phase. The above studies show that phase, temperature, strain amplitude, and waveform changes all significantly affect thermomechanical fatigue deformation mechanisms. Although some researchers have conducted preliminary explorations of thermomechanical fatigue behavior of Ni-based single crystal superalloys, the plastic deformation mechanisms during fatigue and the processes of crack initiation and propagation are far from being fully understood.

After decades of development, Ni-based single crystal superalloys have made important progress in microstructural stability and mechanical behavior. Although researchers have tried to add some precious metal elements (such as Ru, etc.) to improve microstructural stability and have achieved certain research progress, they still cannot significantly delay the γ' phase rafting process or completely suppress TCP phase precipitation, and the mechanical properties of alloys have not been fully exploited. From the perspective of alloy application, future research on microstructural stability and mechanical properties of Ni-based single crystal superalloys should focus on the following aspects:

(1) Improve alloy microstructural stability. Optimize and adjust existing alloy compositions or explore the addition of new elements (including non-metallic elements) to further improve alloy high-temperature microstructural stability.

(2) Continue to pursue higher creep strength. By studying microstructural changes of existing alloys under high-temperature or ultra-high-temperature creep conditions, explore deformation mechanisms that can maintain high-temperature strength, laying a good foundation for designing alloys with superior creep resistance.

(3) Conduct in-depth research on fatigue failure mechanisms of Ni-based single crystal superalloys. The fatigue process of superalloys is very complex, with cracks initiating in different ways and locations under different conditions, making it difficult to establish a unified fracture failure model to describe this process. Therefore, more work is needed to explore the relationship between microstructural evolution and fatigue fracture.

References

[1] Reed R C. The Superalloys Fundamentals and Applications. Cambridge: Cambridge University Press, 2006: 19 [2] Hu Z Q, Liu L R, Jin T, Sun X F. Aeroengine, 2005; 31(3): 1 (胡壮麒, 刘丽荣, 金涛, 孙晓峰. 航空发动机, 2005; 31(3): 1) [3] Sun X F, Jin T, Zhou Y Z, Hu Z Q. Mater China, 2012; 31(12): 1 [4]

Darolia R, Lahrman D F, Field R D, Sisson R. In: Antolovich S D, Stusrud R W, MacKay R A, Anton D L, Khan T, Kissinger R D, Klarstrom D L eds., Superalloys 1988, Champion, PA: TMS, 1988: 255 [5] Tan X P, Liu J L, Jin T, Hu Z Q, Hong H U, Choi B G, Kim I S, Jo C Y. Philos Mag Lett, 2012; 92: 556 [6] Tan X P, Liu J L, Jin T, Hu Z Q, Hong H U, Choi B G, Kim I S, Yoo Y S, Jo C Y. Metall Mater Trans, 2012; 43A: 3608 [7] Tan X P, Hong H U, Choi B G, Kim I S, Jo C Y, Jin T, Hu Z Q. J Mater Sci, 2013; 48: 1085 [8] Argence D, Vernault C, Desvallées Y, Fournier D. In: Pollock T M, Kissinger R D, Bowman R R, Green K A, McLean M, Olson S, Schirra J J eds., Superalloys 2000, Champion, PA: TMS, 2000: 829 [9] Zhang J X, Koizumi Y, Kobayashi T, Murakumo T, Harada H. Metall Mater Trans, 2004; 35A: 1911 [10] Wang X G, Liu J L, Jin T, Sun X F. Mater Sci Eng, 2014; A598: 1 [11] Wang X G, Liu J L, Jin T, Sun X F, Hu Z Q, Do J H, Choi B G, Kim I S, Jo C Y. Mater Des, 2015; 67: 543 [12] Murakumo T, Kobayashi T, Koizumi Y, Harada H. Acta Mater, 2004; 52: 3737 [13] Mughrabi H. Mater Sci Technol, 2009; 25: 191 [14] Pollock T M, Argon A S. Acta Metall, 1994; 42: 1859 [15] Wang X G. PhD Dissertation, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, 2015 (王新广. 中国科学院金属研究所博士学位论文, 沈阳, 2015) [16] Tan X P, Liu J L, Jin T, Hu Z Q, Hong H U, Choi B G, Kim I S, Jo C Y. Mater Sci Eng, 2013; A580: 21 [17] Wang X G, Liu J L, Jin T, Sun X F, Zhou Y Z, Hu Z Q, Do J H, Choi B G, Kim I S, Jo C Y. Adv Eng Mater, 2015; 17: 1034 [18] Rae C M F, Reed R C. Acta Mater, 2001; 49: 4113 [19] Seiser B, Drautz R, Pettifor D G. Acta Mater, 2011; 59: 749 [20] Yeh A C, Tin S. Metall Mater Trans, 2006; 37A: 2621 [21] Hobbs R A, Zhang L, Rae C M F, Tin S. Metall Mater Trans, 2008; 39A: 1014 [22] Ofori A P, Humphreys C J, Tin S, Jones C N. In: Green K A, Pollock T M, Harada H, Howson T E, Reed R C, Schirra J J, Walston S eds., Superalloys 2004, Champion, PA: TMS, 2004: 787 [23] Sato A, Harada H, Yokokawa T, Murakumo T, Koizumi Y, Kobayashi T, Imai H. Scr Mater, 2006; 54: 1679 [24] Gao S, Zhou Y Z, Li C F, Cui J P, Liu Z Q, Jin T. J Alloys Compd, 2014; 610: 589 [25] Cheng K Y. PhD Dissertation, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, 2009 (成魁宇. 中国科学院金属研究所博士学位论文, 沈阳, 2009) [26] Tan X P. PhD Dissertation, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, 2012 (谭喜鹏. 中国科学院金属研究所博士学位论文, 沈阳, 2012) [27] Luo Y P. Master Thesis, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, 2014 (罗银屏. 中国科学院金属研究所硕士学位论文, 沈阳, 2014) [28] Chen J Y, Feng Q, Sun Z Q. Scr Mater, 2010; 63: 795 [29] Pollock T M, Argon A S. Acta Metall, 1992; 40: 1 [30] Field R D, Pollock T M, Murphy W H. In: Antolovich S D, Stusrud R W, MacKay R A, Anton D L, Khan T, Kissinger R D, Klarstrom D L eds., Superalloys 1992, Champion, PA: TMS, 1992: 557 [31] Zhang J X, Murakumo T, Koizumi Y, Kobayashi T, Harada H, Masaki S. Metall Mater Trans, 2002; 33A: 3741 [32] Huang M, Cheng Z Y, Xiong J C, Li J R, Hu J Q, Liu Z L, Zhu J. Acta Mater, 2014; 76: 294 [33] Gabrisch H, Mukherji D. Acta Mater, 2000; 48: 3157 [34] Wang X G, Liu J L, Jin T, Sun X F, Zhou Y Z, Hu Z Q, Do J H, Choi B G, Kim I S, Jo C Y. Mater Sci Eng, 2015; A626: 406 [35] Eggeler G, Dlouhy A. Acta Mater, 1997; 45: 4251 [36] Srinivasan R, Eggeler G F, Mills M J. Acta Mater, 2000; 48: 4867

[37] Zhang J X, Murakumo T, Koizumi Y, Kobayashi T, Harada H. Acta Mater, 2003; 51: 5073 [38] Reed R C, Cox D C, Rae C M F. Mater Sci Eng, 2007; A448: 88 [39] le Graverend J B, Cormier J, Jouiad M, Gallerneau F, Paulmier P, Hanmon F. Mater Sci Eng, 2010; A527: 5295 [40] Caron P. In: Pollock T M, Kissinger R D, Bowman R R, Green K A, McLean M, Olson S, Schirra J J eds., Superalloys 2000, Champion, PA: TMS, 2000: 737 [41] Li Q Q. Master Thesis, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, 2012 (李芹芹. 中国科学院金属研究所硕士学位论文, 沈阳, 2012) [42] Li P, Li Q Q, Jin T, Zhou Y Z, Li J G, Sun X F, Zhang Z F. Int J Fatigue, 2014; 63: 137 [43] Auerswald J, Mukherji D, Chen W, Wahi R P. Z Metallkd, 1997; 88: 652 [44] Wang X G, Liu J L, Jin T, Sun X F, Zhou Y Z, Hu Z Q, Do J H, Choi B G, Kim I S, Jo C Y. Scr Mater, 2015; 99: 57 [45] Zhang X. PhD Dissertation, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, 2006 (张炫. 中国科学院金属研究所博士学位论文, 沈阳, 2006) [46] Gabb T P, Welsch G E. Scr Metall, 1986; 20: 1049 [47] Gabb T P, Welsch G E. Acta Metall, 1989; 37: 2507 [48] Milligan W W, Jayaraman N. Mater Sci Eng, 1986; 82: 127 [49] Zhou H, Ro Y, Harada H, Aoki Y, Arai M. Mater Sci Eng, 2004; A381: 20 [50] Han G M. Master Thesis, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, 2010 (韩国明. 中国科学院金属研究所硕士学位论文, 沈阳, 2010) [51] Kraft S, Zauter R, Mughrabi H. Fatigue Fract Eng Mater Struct, 1993; 16: 237 [52] Liu F, Wang Z G, Ai S H, Wang Y C, Sun X F, Jin T, Guan H R. Scr Mater, 2003; 48: 1265 [53] Zhang J X, Harada H, Ro Y, Koizumi Y, Kobayashi T. Acta Mater, 2008; 56: 2975 [54] Moverare J J, Johansson S, Reed R R. Acta Mater, 2009; 57: 2266 [55] Zhang J. PhD Dissertation, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, 2006 (张剑. 中国科学院金属研究所博士学位论文, 沈阳, 2006) [56] Xu Y B. Master Thesis, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, 2013 (徐砚博. 中国科学院金属研究所硕士学位论文, 沈阳, 2013)

Note: Figure translations are in progress. See original paper for figures.