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LETTER

Effect of γ′ Size on Intermediate Temperature Stress Rupture Property of the Third Generation Single Crystal Nickel-base Superalloy Containing Re

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Abstract: Three kinds of γ' phases with different morphologies and sizes were obtained by changing the primary aging temperature (1150, 1180 and 1200 °C) in the heat treatment process. Then, the samples with different γ' morphologies were tested under condition of intermediate temperature and high stress (760 °C/800 MPa). The results show that the size of γ' phase plays an important role in the stress rupture property. Increasing the size of γ' phase appropriately can promote the uniform deformation of the alloy, thus improving the stress rupture life of the alloy.

Key words: single crystal superalloy; heat treatment; microstructure; stress rupture property

Since the first generation of nickel base single crystal superalloy was successfully developed, people have been committed to developing a new generation of superalloy that can withstand higher service temperature[1]. The addition of 3% Re element has made a breakthrough progress, by which the temperature bearing capacity of the second generation single crystal alloy is increased by 30 °C and the creep life at intermediate temperature is increased by more than two times compared to the first generation superalloy[2-4]. However, with the increase of Re content, the creep properties of the third generation single crystal superalloy are improved at high temperature, while the creep properties at intermediate temperature are not as good as those of the second generation single crystal alloy[5]. In fact, higher generation single crystal superalloys (such as the fourth generation) have both higher intermediate and high temperature creep resistance due to the addition of Ru[6-8]. In the past, dislocation processes of creep regime have been thoroughly investigated. In the high temperature and low stress regime, tertiary creep is prevalent, with little primary creep (less than 1%), a large initial creep rate, and then a rapid decrease[9,10]. The significant morphological change at primary stage is rafting[11,12], that is, the precipitate morphology transforms from cuboidal to irregular, elongated plates, which is driven by the applied stress and misfit stress[13]. The creep rate decreases gradually with strong strain hardening in the γ channel. Primary creep is followed by a long-term low creep rate which is nearly constant[14]. Under high stresses, the γ' phase can be shared by couples of $a/2\langle 011\rangle$ dislocations, which is the classical mechanism of high temperature γ' cutting[14,15].

The conditions of intermediate temperature (650~850 °C) and high stress (more than 500 MPa) have been reported to be more complicated. For example, there is no significant rafting and the primary creep strain of single crystal superalloy is usually high[16]. Rae et al[17] found that the larger creep strain in the primary stage is usually related to the occurrence of $a\langle 112\rangle$ dislocation ribbons, which can cut through both γ and γ' phases simultaneously. The secondary creep rate is closely related to the primary strain[17,18]. These dislocation ribbons are generated from $a/2\langle 110\rangle$ dislocations in the γ matrix at the early stage in the deformation[18]. Sufficient dislocations with suitable Burgers vector nucleate the ribbons, but too many dislocations cannot propagate, which is the so-called "Rae window"[18]. When the shear stress is large enough, they will

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decompose into a<112> and cut into the interior of the γ' phase. If only a single dislocation cuts into the interior of the γ' phase, stacking faults will appear, which is also called "stacking fault shearing"; if the γ' phase is cut into the form of dislocation pairs, anti-phase boundary will be formed[16,17,19,20]. Recently, Wu et al[21] reported the nucleation of planar faults and provided a better understanding of the processes in the early stages during low temperature and high stress creep of single crystal superalloys. Intermediate temperature creep usually takes place at the bottom of blade (blade root, platform) and the lowest part of airfoil. In addition, the complex cooling channel in blade will also produce thermal stress, and the working temperature of these parts is about 760 °C[5]. Compared with the high temperature creep, the intermediate temperature creep attracts less attention, but for the development of a new alloy, both are indispensable. The deformation mechanism of intermediate temperature creep is very clear, but how to improve the intermediate temperature creep strength of the alloy on the premise of maintaining good microstructure stability is still an important topic in the development of the third generation single crystal alloy.

The experimental third generation single crystal nickel-base superalloy was used in this study. Three morphologies of γ' phase were obtained in different samples by adjusting the primary aging temperature. The purpose of this study is to better understand the relationship between the γ' precipitate size and stress rupture life at intermediate temperatures.

1 Experiment

The nominal compositions of the single crystal nickel-base superalloy with Re are shown in Table 1. The master alloy was melted in ZG-0.01 vacuum induction furnace, and the single crystal rod with [001] orientation was prepared by screw selecting method, with the misorientation from [001] within 10°. The diameter of the single crystal rod was 16 mm, the length was 210 mm, and the casting speed was 6 mm/min. Considering that the alloy used in this work contains more Re elements, two-stage solid solution and two times aging heat treatment were selected to better eliminate the element segregation in the alloy. Solution treatment can eliminate dendrite segregation during solidification to a great extent, so as to eliminate eutectic and make γ' precipitates more uniform. Primary aging was used to adjust the morphology and size of γ' precipitates and secondary aging can further promote the diffusion of elements[22]. Only the primary aging temperature was changed to obtain different γ' precipitate sizes. The specific heat treatment steps are shown in Table 2. After heat treatment, 10 and 67 mm of the single crystal rods were cut off to observe the microstructure and machined into stress rupture specimens. In this experiment, F50 field emission scanning electron microscope (SEM) was used to observe the γ/γ' microstructures. A JEOL JEM-2100F (200 kV) transmission electron microscope (TEM) was used to observe dislocation configuration after deformation. Copper sulfate solution (20 mL HCl+5 g $CuSO_4$+25 mL H_2O) was used to etch the SEM and metallographic microstructures. A solution of 10% perchloric acid in alcohol was used for electrolytic etching of TEM samples at −20 °C. The size of γ' phase was calculated by the software of Image-Pro plus.

Table 1 Normal chemical composition of the single crystal nickel-base superalloy (wt%)

Co	Cr	Mo+W+Ta	Al	Re	Hf	C	Ni
12	3	15	5.9	5	0.1	0.02	Bal.

Table 2 Heat treatment processes for different samples (AC: air cooling)

Sample	Solution	Primary ageing	Second ageing
H1	1330 °C/16 h+ 1340 °C/16 h AC	1150 °C/4 h AC	870 °C/24 h AC
H2	Same as H1	1180 °C/4 h AC	Same as H1
H3	Same as H1	1200 °C/4 h AC	Same as H1

2 Results and Discussion

2.1 γ/γ' microstructures after heat treatment

The microstructure of the alloy under different heat treatment conditions is shown in Fig.1. It can be seen that with the increase of primary aging temperature, the γ' size increases and the alignment becomes better. The statistical histogram of the γ' size is shown in Fig.2. It is necessary to point out that the fine secondary γ' phase is found in γ channels in all samples, and a small amount of carbide is precipitated in the interdendritic regions.

2.2 Stress rupture properties

Fig.3 shows the stress rupture life of the alloy at 760 °C and 800 MPa. It can be seen that the rupture life of H2 sample is much longer than that of the other two alloys. Obviously, increasing the γ' size can significantly affect the stress rupture life.

Fig. 4 shows different morphologies of the samples after fracture failure. It can be seen that there are great differences in the macro fracture of the three samples. The fracture of H2 sample is composed of multiple {111} planes intersecting each other, while the fracture of the other two samples is mainly broken along a slip plane (Fig. 4a). In addition, the deformation of H2 sample is much more uniform. However, the deformation of H1 and H3 sample severely occurs non-uniformly, and the cross section is elliptical. The minor axis of the ellipse is parallel to the <110> direction, which is recognized from the dendrite pattern (Fig.4b). The major axis and minor axis of the sample are represented by a and b respectively. The ratios of a to b in the H1, H2 and H3 samples are 1.35, 1.03 and 1.36, respectively (Fig. 4c). It is obvious that the longer the life of the sample, the smaller the corresponding ratio. The ellipticity of the specimen is the result of less activation of the slip systems, and there is a negative correlation between the degree of ellipticity and the life of the specimen. The deformation mechanism of the alloy will be discussed in detail.

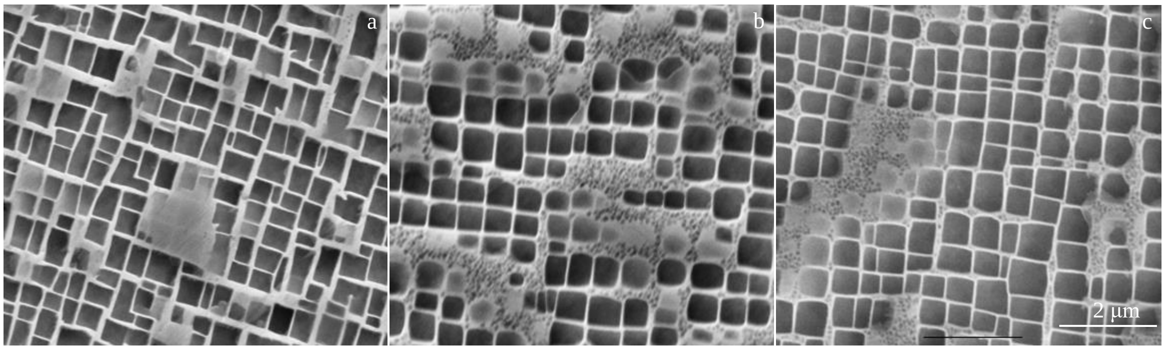

Fig.1 Morphologies of γ' precipitates in different samples: (a) H1, (b) H2, and (c) H3

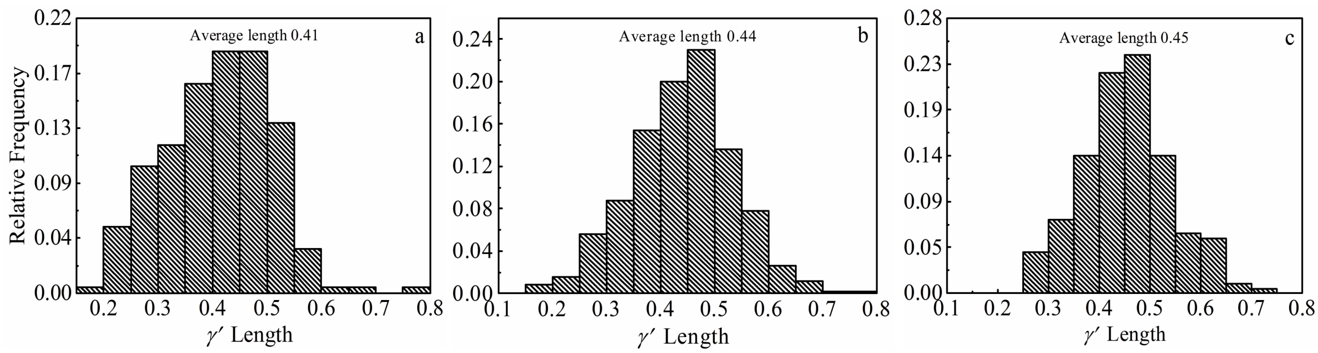

Fig.2 Distribution of γ' length in different samples: (a) H1, (b) H2, and (c) H3

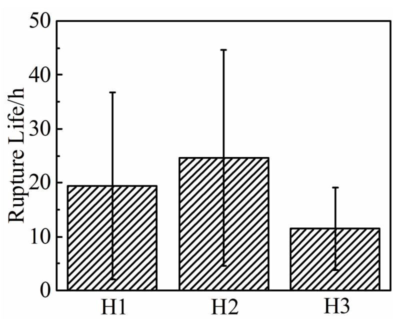

Fig.3 Stress rupture life of different samples

Fig. 5 shows the typical dislocation configuration observed after rupture. It can be seen that the dislocation density is high, and the dislocation arrangement is disordered. A large number of stacking faults can be clearly seen in γ' phase, which is the main cause of alloy failure. It is known that the creep curve can be divided into three stages, and the deformation mechanism in Re-containing single crystal superalloy when deformed at intermediate temperature is the dislocation slip (or cross-slip) and the γ'-cutting by stacking fault[5,16,23].

It has been mentioned that the primary stage of intermediate temperature creep plays a very important role in the whole deformation process. In this stage, <110> {111} matrix dislocations release the misfit stress and the stable dislocation network is formed, which has little contribution to the creep strain. However, <112> {111} provides the main contribution to the creep strain, which can continuously pass through multiple γ' precipitates, resulting in a creep strain 13~40 times greater than the former[17]. In fact, at 760 °C, there is a competition between the Orowan mechanism and the γ' cutting mechanism by <112> ribbons[5]. The Orowan resistance increases due to the narrow γ channel, which makes it difficult for dislocations to bow out and can only be confined to local channels. Therefore, it not only restricts the interaction between dislocations, but also promotes the generation and movement of <112> {111} ribbons, thereby delaying the transition from the primary creep stage to the second stage and increasing the primary creep strain and inhomogeneous deformation. Consequently, properly increasing the size of the γ' precipitates and the width of the γ channels can promote <110> {111} slip in the matrix, thus forming a uniform and regular dislocation morphology, which is conducive to reducing the creep strain. However, if the γ' size is too large, the bypass mechanism of dislocations controlled by the Orowan stress will be greatly increased, which will significantly increase the creep rate of the second stage, and reduce the creep life of the alloy[5].

Obviously, adjusting the morphology of γ' phase alone is not enough to fundamentally address the unsatisfactory intermediate temperature creep property of the third generation single crystal superalloy, which is determined by the limitations of the alloy composition. Therefore, to greatly

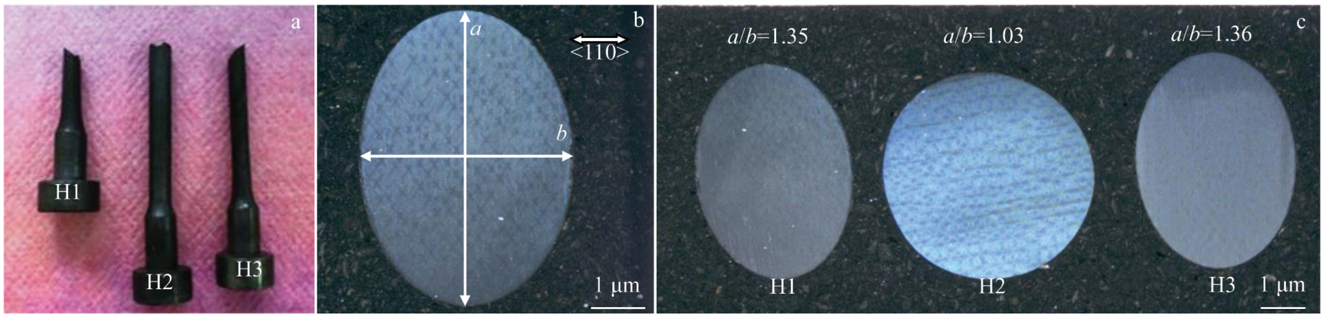

Fig.4 Macroscopical fracture morphology (a) and cross-sectional metallographic images of fracture surface (b, c) of different samples (high *a/b* value means uneven deformation)

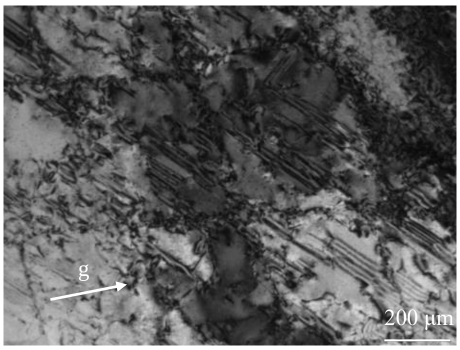

Fig.5 TEM image of deformed microstructure in H1 sample after stress rupture at 760 °C and 800 MPa

improve the intermediate temperature creep property, it is necessary to further optimize the composition of the alloy.

3 Conclusions

1) The effect of γ' size on intermediate temperature stress rupture property can be investigated by changing the primary aging temperature (1150, 1180 and 1200 °C) of a Re-containing single crystal superalloy. γ' size increases with the increase of primary aging temperature.

2) When the primary aging temperature is 1180 °C, the alloy shows the best stress rupture life. Increasing the γ' size properly can improve the stress rupture property.

3) The deformation degree is directly related to the life of the alloy. When the specimen has serious uneven deformation, the cross section is elliptical, and the life of the alloy is usually shorter. When the deformation is uniform, the life time is usually longer.

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γ'相尺寸对一种含Re单晶镍基高温合金中温持久性能的影响

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摘 要：通过改变含Re单晶镍基高温合金热处理过程中的一次时效温度（1150， 1180和1200 ℃），获得了3种不同形貌和尺寸的γ'相。随后，对含有不同γ'形貌的3种样品进行了中温高应力（760 ℃/800 MPa）持久试验。结果表明，γ'相的大小对单晶高温合金的持久性能有很大影响。适当增大γ'相的尺寸可以促进合金的均匀变形，从而提高合金的中温持久寿命。

关键词：单晶高温合金；热处理；显微组织；持久

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