Effect of Mo content on microstructure and stress-rupture properties of a Ni-base single crystal superalloy

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Abstract

The additional 1.5 wt% Mo was added in a Ni-base single crystal (SC) alloy with the composition of Ni–6.5Al–8.0Mo–2.4Cr–6.2Ta–4.9Co–1.5Re–(0.01–0.05)Y (wt%) to study the effect of Mo content on the microstructure and stress-rupture properties. The creep and stress-rupture tests under the conditions of 850°C/500 MPa and 1100°C/130 MPa were conducted, and the microstructure of as-cast, heat treated and stress ruptured specimens were analyzed. It was found that the 1.5 wt% Mo addition enhanced the stress-rupture lives at both intermediate (850°C) and high (1100°C) temperatures. The microstructure analysis showed that adding 1.5 wt% Mo in the basic alloy affected the microstructure dramatically, i.e., the Mo-rich phases formed in the specimens of as-cast and stress-ruptured specimens. It is considered that the improvement of the stress-rupture lives is due to the strengthening effect of Mo to both \( \gamma \) and \( \gamma' \) phases and the decrease of stacking fault energy, diffusion constant and dislocation spacing. The Mo-rich phases precipitated under condition of 1100°C/130 MPa did not affect the creep and stress-rupture properties obviously in the present study.

Keywords: Ni-base single crystal superalloy; Mo content; Stress-rupture property

1. Introduction

Ni-base single crystal (SC) superalloy is widely used for modern aero-engines due to its excellent comprehensive properties [1,2]. In order to optimize and improve the high temperature creep resistance of SC superalloys, refractory elements such as rhenium (Re) or tungsten (W) were added in SC superalloys. For example [3], the refractory elements (i.e. W + Mo + Re + Ta) contents of representative first generation alloy (CMSX-2) is about 14 wt% while in the third generation alloy (CMSX-10) it is greater than 20 wt%. However the disadvantage of high density is obvious.

It should be noted that Mo has a much lower density compared to other refractory elements. Therefore, using Mo as the main solution strengthening element will decrease the density of SC superalloy, which undoubtedly expands the application of Mo-strengthening SC superalloy. For example, LDS-5555 (9.5 wt% Mo) [4], a new low density Ni-based SC superalloy was developed by NASA. Recently, a high Mo content Ni3Al alloy IC21 [5] was reported to have good high temperature properties and low density and cost.

However, it was found that the Mo content should be optimized to achieve the best mechanical properties of SC superalloy [6,7]. Excessive Mo addition may precipitate deleterious third phase and degrade the mechanical properties. Recently the research on the effect of Mo on microstructure and mechanical property of Ni-based SC alloys is very limited. In the present study, the influence of Mo content on the microstructure and stress-rupture properties of a low density Ni-based SC alloy was investigated. The target of this research is to provide references for the future alloy composition adjustment on the alloy.

2. Materials and experimental

Two single crystal superalloys were used in the present investigation with the nominal composition (wt%) of
Table 1
Heat treatment schedules of the two alloys.

<table>
<thead>
<tr>
<th>Solution treatment</th>
<th>Aging treatment</th>
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<tbody>
<tr>
<td>Alloy 1 1310 °C/2 h +1315 °C/2 h +1320 °C/6 h + 1325 °C/6 h, FAC (Flow air cooling)</td>
<td>1080 °C/2 h, AC (Air cooling) + 871 °C/32 h, AC</td>
</tr>
<tr>
<td>Alloy 2 1300 °C/2 h +1305 °C/2 h +1310 °C/6 h + 1315 °C/6 h, FAC</td>
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Ni–6.5Al–8.0Mo–2.4Cr–6.2Ta–4.9Co–1.5Re–(0.01–0.05)Y (Alloy 1) and Ni–6.5Al–9.5Mo–2.4Cr–6.2Ta–4.9Co–1.5Re–(0.01–0.05)Y(Alloy 2 with 1.5% higher Mo content), respectively.

The master alloys were prepared by vacuum induction melting. A high-rate-solidification (HRS) Bridgman apparatus was used to cast the SC rods (φ15*150 mm) with the withdraw rate 4 mm/min. The as-cast rods were etched with 500 ml H2O2 and 4500 ml HCl for 10 min to confirm the single crystal structure of the rods. Laue back-reflection method was used to measure the orientation. Only specimens with the orientations within 15° deviating from <001> were used for the present study. The heat treatment schedules for the two alloys are shown in Table 1.

The specimens with the gauge length of 25 mm and diameter of 5 mm cut and machined from the heat treated SC rods were used for the creep and stress-rupture tests. The creep and stress-rupture tests were carried out on creep testing machines in ambient atmosphere under the test conditions of 850 °C/500 MPa and 1100 °C/130 MPa.

The optical microscopy (OM), scanning electron microscopy (SEM) with energy dispersive spectrometer (EDS) were used for the microstructure analysis. An X-ray diffractometer (XRD) was adopted to measure the γ/γ’ lattice misfit of the alloys. A JEM2000 was used for TEM observation.

The longitudinal section microstructure of the alloys after stress rupture tests was observed by SEM. All the specimens for observation were taken at the positions of 10 mm to the fracture surface.

3. Results and discussion

3.1. Microstructure of as-cast and heat treatment alloys

The as-cast microstructure is typically shown in Fig. 1. It was found that the similar phases existed in interdendritic precipitation region of Alloy 1 and Alloy 2. The compositions of different phases were analyzed, and the result indicated that the large black phases are γ’ phase. Phase A and phase B are found adjacent to the large γ’ phases and are both eutectic. The white phases (in phases A and B) are rich in Mo, and the needles like phase C was found in the Mo-rich γ phases region. This precipitation morphology is significantly different from that of other Ni based SC superalloys, where the typical γ/γ’ eutectic microstructure usually exists in the interdendritic region [8,9].

In a recent research [10] on a Mo-rich Ni3Al based single crystal superalloy, the interdendritic precipitation region were analyzed in detail, indicating that the interdendritic precipitation region are mainly large γ’ phases, α-Mo and γ/NiMo eutectic. In this research, however, no α-Mo phases were found since the Mo concentration is not high enough. Therefore it may be considered that the interdendritic precipitation phases (IDP) are mainly large γ’ phases and some Mo-rich eutectic.

It was found that the main difference between the two alloys is the amount of the IDP, i.e., the area fractions of IDP were about 0.16% in Alloy 1 and about 0.50% in Alloy 2, respectively. The reason is that the additional Mo addition decreases the saturation ability of Al atoms in γ matrix phases. The Al atoms gradually enriched in liquid phase during the solidification process and resulted in the precipitation of large γ’ phases, and hence Mo partitions strongly to the γ phase, i.e., the residual liquid is rich in Mo atoms after the precipitation of the large γ’ phases, and leads to the precipitation of Mo-rich phases. The Mo-rich eutectic also had lower melting temperature and would be the source of incipient melting. It is believed that the higher area fraction of IDP will decrease the incipient melting temperatures (IMT) of the alloy. The IMT tests showed that the IMT for Alloy 1 was 1325 °C and 1315 °C for Alloy 2, which is consistent with the as-cast IDP analysis result. Phase A and B were observed in both alloys. However, phase C was barely observed in Alloy 1. Phase C was usually found in the Mo-rich γ phase region rather than adjacent to the large γ’ phases like phase A and B. This may be attributed to the fact that in the higher Mo content in Alloy 2. Mo atoms were enriched in the interdendritic area, and the high Mo concentration of γ phases in interdendritic areas promoted the precipitation of Phase C. The Mo concentration in Alloy 1 is not high enough, and hence Phase C was barely observed.

The microstructure of the two alloys after full heat treatment (shown in Table 1) is demonstrated in Fig. 2. The IDP totally disappeared after the solution treatment. No Mo-rich phases were found in the heat treated alloys. The two alloys have typical Ni-based superalloy microstructure: the γ’ precipitated phases and the γ matrix around it. The average γ’ sizes of the two alloys are almost the same, about 0.44 μm.

3.2. Creep and stress-rupture properties

The creep and stress-rupture tests for two alloys were conducted under the test conditions of 850 °C/500 MPa and
1100 °C/130 MPa. Table 2 lists the stress-rupture lives of the two alloys with the Mo contents of 8.0 wt% (Alloy 1) and 9.5 wt% (Alloy 2). Each value in Table 2 shows the average data of stress rupture life of two specimens under the same test condition.

It can be found that the alloy with 9.5% Mo had longer stress rupture lives than the alloy with 8% Mo under the same test condition. The stress rupture lives increased by 24% under the test condition of 850 °C/500 MPa, and increased by 66% under the test condition of 1100 °C/130 MPa due to the additional 1.5% Mo addition. This indicates that Mo is an effective strengthening element, especially at elevated high temperature service condition. The typical creep curves of the two alloys with the Mo contents of 8.0 wt% (Alloy 1) and 9.5 wt% (Alloy 2) are shown in Fig. 3.

The results in Fig. 3 show that the steady state creep rates, which is usually considered as determining factor for creep resistance, of higher Mo content (Alloy 2) are significantly lower than that of Alloy 1, especially at high temperature. This may be attributed to the following reasons. The previous study [11] revealed that Mo has lower diffusion coefficient compared with other alloying elements, and therefore the addition of Mo can lower the coarsening rate of γ phases of Ni base superalloys. This was confirmed by microstructure analysis of stress ruptured specimens, as shown in Figs. 4 and 5. Fig. 4 shows second electron images (SEI) of the specimens under the test condition of 850 °C/500 MPa for 356.8 h (Alloy 1) and 428.5 h (Alloy 2). Fig. 5 shows backscattered electron images (BSEI) of the specimens under the test condition of 1100 °C/130 MPa for 180.8 h (Alloy 1) and 310.6 h (Alloy 2).

It can be observed that no obvious size deference of γ′ phase has been found between the two alloys although the test time of Alloy 2 was 72 h (20% of total creep life) longer than Alloy 1 at 850 °C/500 MPa, and 130 h (70% of total creep life) longer than Alloy 1 at 1100 °C/130 MPa. This indicates that the coarsening rate of γ′ phases in the 9.5% Mo content alloy was slower than that in 8.0% Mo content alloy. It is also reported [7,12] that the Mo addition can low the stacking fault energy (SFE) in a Ni–Mo binary alloy, and low SFE would reduce the dislocation cross slip and climb since the dissociated a/2 < 110 > dislocations’ mobility was enhanced. The more regular γ′ rafts and lower SEF make Alloy 2 has a lower second stage creep rate than Alloy 1.

Fig. 6 shows the TEM images of the specimens stress ruptured under the test condition of 1100 °C/130 MPa, showing that the dislocation networks formed in the interface of γ/γ′ phases. Table 3 shows the lattice misfit at ambient temperature and dislocation spacing of two alloys. It has been found that the addition of 1.5 wt% Mo leads to a more negative misfit in Alloy 2 and decreases the dislocation spacing. The previous researches of Zhang et al. [13,14] indicates that a larger magnitude of misfit would form a denser dislocation networks in the γ/γ′ interfaces and lower the minimum creep rates during high temperature creep tests. Even though there were small amount of Mo-rich phases precipitation in Alloy 2.
stress-rupture tests. Although the formation of Mo-rich phases could decrease the strengthen effect of Mo, the small amount and dispersed particle phase does not affect the creep and stress-rupture property under 1100 °C/130 MPa, obviously.

4. Conclusions

1. Large γ’ phases and Mo-rich phases rather than typical γ/γ’ eutectic microstructure have been found at the interdendritic region of as-cast alloys. The Mo addition increases the primary γ’ fraction and decreases the IMT.
2. γ’ rafts perpendicular to the applied stress have been found in both alloys at 850 °C/500 MPa and 1100 °C/130 MPa after failure. The lower coarsen rate due to the addition of Mo makes Alloy 2 has more regular γ’ rafts.
3. The additional addition of 1.5 wt% Mo increases the stress-rupture properties. This is attributed to the strengthening of the γ/γ’ phases by Mo and decrease the stacking fault energy, diffusion constant and dislocation spacing. Mo-rich
phase found in Alloy-2 in the crept specimens at 1100 °C/130 MPa, which does not influence the creep and stress-rupture properties obviously during the test condition of 1100 °C/130 MPa.

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References