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Microstructure and Property Stability of Powder Metallurgy Nickel-based U720Li Superalloy During Long-Term Aging

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Abstract: The microstructure evolution and variation of mechanical properties of nickel-based U720Li superalloy were investigated during long-term aging (3000 h) at 680, 700, and 730 °C. The changes in size and morphology of γ' phase were determined. Results show that the coarsening behavior of γ' phase is controlled by the diffusion process. The tensile and creep properties of U720Li superalloy remain stable during the long-term aging at 680 and 700 °C. However, the yield strength gradually decreases and the ductility increases after long-term aging at 730 °C for 500 h. The fatigue and creep properties of U720Li superalloy decreases sharply after long-term aging at 730 °C, which is mainly related to the morphology evolution of γ' phase.

Key words: U720Li; long-term aging; microstructure stability; γ' phase

The superalloys after powder metallurgy (P/M) have been widely used to manufacture gas turbine discs, compressor discs, and other hot end parts, because of their homogenized microstructure, controllable macro-segregation, and excellent high-temperature strength, creep property, and fatigue resistance^[1-6]. With the development of advanced turbine engines, the long-term service under tough conditions is more and more crucial for P/M superalloys^[7-9]. The γ' phase can cause precipitation strengthening effects. However, the size, distribution, and morphology of γ' particles can change during the long-term service at high temperature^[10-14], which may lead to the degeneration of properties. Hence, the investigation of effects of long-term aging on the microstructure evolution and property variation is essential.

The U720Li superalloy is mainly cast or forged and then used to manufacture turbine discs for advanced engines due to its excellent high-temperature properties and damage tolerance^[15-19]. However, the components of U720Li superalloy manufactured by P/M routes are rarely investigated. Therefore, the reliability and durability of P/M U720Li superalloy during long-term service were studied in this research. The microstructure evolution and property stability

of U720Li superalloy were investigated at different aging temperatures of 680, 700, and 730 $^{\circ}$ C.

1 Experiment

The composition of U720Li superalloy powder is listed in Table 1. The powder was prepared by argon atomization and then selected. Afterwards, the powder was processed by hot isostatic pressing (HIP), hot extrusion, isothermal forging, and heat treatment at sub-solvus temperature. Then, the powder suffered the two-step aging to achieve a homogenized structure: 760 °C/16 h/air cooling (AC) and 650 °C/24 h/AC. Finally, the specimens were machined and aged at 680, 700, and 730 °C, separately. The aging duration ranged from 500 h to 3000 h.

The tensile, creep, and fatigue properties of P/M U720Li superalloys after heat treatment and long-term aging were investigated. The tensile properties were tested at 650 °C; the

 Table 1
 Composition of P/M U720Li superalloys (wt%)

Al	В	С	Со	Cr	Mo	Si	Ti	W	Ni
2.5	0.011	0.014	15	16	3.0	0.05	5.0	1.25	Bal.

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creep properties were studied at 625 °C/730 MPa to obtain the elongation after aging for 100 h; the fatigue life at 650 °C/1250 MPa under the strain amplitude of 0.05 and loading frequency of 0.33 Hz was also obtained. The specimens were prepared by mechanical grinding, polishing, and electrochemical polishing for microstructure observation. The field emission scanning electron microscopy (SEM, ZEISS SUPRA 55) was used.

2 Results and Discussion

2.1 Microstructure evolution during long-term aging

The γ' phase (Ni₃Al) plays a major role in precipitation strengthening of U720Li superalloy. As shown in Fig.1, before long-term aging, three types of γ' phases can be observed. The large primary γ' phase with 1~3 µm in diameter is mainly distributed along the grain boundary. The size of flower-like or spherical secondary γ' phase ranges from 100 nm to 250 nm, and the average size is 121.6 nm. The secondary γ' phase is generally located in the grains. Moreover, a large number of tertiary γ' particles of 15~30 nm in diameter are scattered among the large γ' phases.

Fig.2 shows the morphologies of γ' particles after long-term aging. Compared with that before long-term aging, the average size of γ' particles after aging at 680, 700, and 730 °C increases to 134.7, 134.9, and 135.4 nm, respectively. Additionally, the shape of secondary γ' phase and the distribution of tertiary γ' particles change significantly. Specifically, the number of tertiary γ' particles is decreased with increasing the aging temperature. The flower-like secondary γ' phase is transformed into spherical, ellipsoidal, or rounded-corner cubic shape, which is mainly caused by the

competition between surface energy and lattice mismatchinduced elastic interaction energy. The change of total free energy ΔU can be determined by Eq.(1)^[12], as follows:

$$\Delta U = \Delta U_{\rm s} + \Delta U_{\rm I} = \gamma_{\rm s} \Delta A - B \Delta V = 2\gamma_{\rm s} V/d - BV \tag{1}$$

where $\Delta U_{\rm s}$ is the surface energy; $\Delta U_{\rm I}$ means the elastic interaction energy; *A* represents the surface area of γ' phase; *V* and *d* are the volume fraction and diameter of γ' phase, respectively; *B* is a constant; $\gamma_{\rm s}$ is the surface tension.

When the surface energy dominates, the decrease of surface energy becomes the driving force of the coarsening of γ' phase. By integrating the coarse secondary γ' phase and tiny tertiary γ' particles, the notch of the flower-like γ' phase is filled and the area of the γ' matrix interface is thereby decreased. As the cubic γ' phase is transformed into the spheric or rounded shape, the specific surface area is decreased. Thus, the total free energy can be deduced and the system becomes more stable. With increasing the γ' size, the contribution of elastic interaction energy to the total free energy becomes more significant, and the coarsening of γ' phase slows down gradually.

In general, the coarsening rate of γ' phase is closely related to the coherent strain, γ/γ' interface energy, and the element diffusion. During the long-term aging, the coarsening of γ' phase obeys the Ostwald ripening theory^[9], indicating that the average particle diameter and the aging duration have a relationship, as follows:

$$\overline{d}^{3} - \overline{d}_{0}^{3} = \frac{64\sigma DC_{e}V_{m}^{2}t}{9RT} = Kt$$
⁽²⁾

where t is the aging time; D is the diffusion coefficient of solute in the matrix; \overline{d}_0 and \overline{d} are the average diameters of γ' particles before and after aging, respectively; σ means the γ/γ'



Fig.1 Morphologies of primary γ' phase (a), secondary γ' phase (b), and tertiary γ' particles (c) in U720Li superalloy before long-term aging



Fig.2 Morphologies of γ' particles after long-term aging at 680 °C (a), 700 °C (b), and 730 °C (c) for 3000 h

interface energy; $C_{\rm e}$ is the atomic fraction of solute in equilibrium with the precipitate; $V_{\rm m}$ represents the molar volume of the precipitate; R is the universal gas constant; T is the absolute temperature; K is the rate constant.

The relationship between the precipitation size and aging time is plotted in Fig.3, which verifies the cube power law. In general, with increasing the aging temperature from 680 °C to 730 °C, the rate constant is increased from 226.2 nm³/h to 233.5 nm³/h, which is caused by the enhanced diffusion rate at higher temperature.

The morphologies of γ' particles after aging at 730 °C for 500, 1000, 2000, and 3000 h are shown in Fig. 4. With prolonging the aging duration, the number of tertiary γ' particles is decreased, and the flower-like secondary γ' phase changes to round shape. Therefore, the increase in aging duration and that in aging temperature have similar effects on the γ' phase evolution.

2.2 Effects of long-term aging on mechanical properties

The tensile properties at 650 $^{\circ}$ C of U720Li superalloy before and after aging under different conditions are shown in



Fig.3 Relationship of precipitate size $(\overline{d}^3 - \overline{d}_0^3)$ and aging time (t) during long-term aging at 680, 700, and 730 °C

Fig. 5. Generally, the yield strength and ultimate tensile strength of U720Li superalloy aged at 680 and 700 ° C decrease slightly, compared with those before long-term aging, indicating that the mechanical properties remain stable at these aging conditions. While the yield strength of U720Li superalloy aged at 730 ° C is decreased significantly with prolonging the aging duration. The decrease of yield strength is caused by the weakened critical resolved shear stress (CRSS) $\Delta \tau$, considering the interaction between the precipitation strengthening particles and the dislocation pairs. Thus, CRSS can be estimated by Eq.(3), as follows:

$$\Delta \tau = 0.86 \frac{\theta V^{\frac{1}{2}} \omega}{|\mathbf{b}| d} \left(1.28 \frac{d\gamma_0}{\omega T} - 1 \right)^{\frac{1}{2}}$$
(3)

where θ is the line tension, which is nearly a constant; *b* means Burgers vector; *d* is the diameter of γ' particles after aging; *V* is the volume fraction of γ' phase; ω is a constant; γ_0 represents the anti-phase boundary energy (APBE). Basically, with increasing the particle size, the value of $\Delta \tau$ is decreased, which thereby causes the strength degeneration. During the long-term aging at 730 °C, the number and volume fraction of fine tertiary γ' particles decease dramatically, and the average diameter of γ' increases, leading to the property degradation.

The ductility change of the aged U720Li superalloy is different, as shown in Fig. 5b. For the specimen aged at 730 °C, the elongation fluctuates and a certain rise in elongation can be observed at the specific aging time. Fig.6 shows the tensile fractographs at 650 °C of U720Li superalloys aged at 730 °C for 500 and 3000 h. The mixed ductile-brittle fracture can be observed in the aged U720Li superalloy, as the dimples, quasi-cleavage facets, intergranular cracks, and ductile intergranular tears exist simultaneously. Compared with the specimen aged for 500 h, the fracture of U720Li superalloy after long-term aging for 3000 h has more deep



Fig.4 Morphologies of γ' particles after aging at 730 °C for 500 h (a), 1000 h (b), 2000 h (c), and 3000 h (d)



Fig.5 Tensile properties at 650 °C of U720Li superalloy under different aging conditions: (a) yield strength and ultimate tensile strength; (b) elongation



Fig.6 Tensile fractographs at 650 °C of U720Li superalloys aged at 730 °C for 500 h (a) and 3000 h (b)



Fig.7 Elongation after creep at 625 °C/730 MPa (a) and fatigue life at 650 °C/1250 MPa with strain amplitude of 0.05 and loading frequency of 0.33 Hz (b) for U720Li superalloys after long-term aging

dimples and ductile intergranular tears, indicating that the ductility dominates under this condition.

The creep resistance of U720Li superalloys at 625 °C/730 MPa for 100 h is shown in Fig.7a; the fatigue life of U720Li superalloy at 650 °C/1250 MPa after long-term aging is shown in Fig.7b. According to Fig.7a, the creep resistance of U720Li superalloys after aging at 680 and 700 °C for 3000 h is relatively stable, while that after aging at 730 °C decreases significantly. Additionally, the fatigue life of all U720Li superalloys aged at 680, 700, and 730 °C is decreased, and the fatigue life of U720Li superalloy aged at 730 °C is the shortest, as shown in Fig.7b. Basically, this phenomenon is mainly caused by the coarsening of secondary γ' phase and the

reduction of fine tertiary γ' phase.

3 Conclusions

1) Three types of γ' phases in U720Li superalloys before long-term aging can be observed, and the shape of secondary γ' phase and the distribution of tertiary γ' particles change significantly after aging. The number of tertiary γ' particles is decreased with increasing the aging temperature, and the flower-like secondary γ' phase is transformed into round shape, which is mainly caused by the competition between the surface energy and elastic interaction energy.

2) The coarsening of γ' phase obeys the Ostwald ripening theory. With increasing the temperature from 680 ° C to

730 °C, the coarsening rate is increased slightly from 226.2 nm³/h to 233.5 nm³/h, which is induced by the enhanced diffusion at higher temperature.

3) The tensile properties and creep resistance of U720Li superalloy after aging at 680 and 700 °C for 3000 h are relatively stable, while those of U720Li superalloy aged at 730 °C decrease significantly, which is mainly caused by the coarsening of secondary γ' phase and the reduction of fine tertiary γ' phase.

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粉末镍基U720Li高温合金长期时效下的组织与性能稳定性

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摘 要:研究了经粉末冶金的镍基U720Li高温合金在680、700和730℃长期时效(3000 h)下的组织演变及力学性能变化,表征了y'相 在长期时效过程中的形貌与尺寸变化。结果表明,y'相粗化行为由扩散过程控制。在680和700℃下的长期时效过程中,U720Li高温合 金的抗拉伸和蠕变性能保持稳定;但在730℃/500 h长期时效后,合金屈服强度逐渐降低,塑性增加。在730℃长期时效过程中, U720Li高温合金的疲劳和蠕变性能急剧下降,这与y'相的形貌演变有关。

关键词: U720Li; 长期时效; 组织稳定性; y'相

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