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Microstructure evolution and creep behaviors of a directionally solidified nickel-base alloy under long-life service condition

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ABSTRACT

By means of creep properties measurement, microstructure observation and lattice parameters measurement, the microstructure evolution and creep behaviors of a directionally solidified nickel-based alloy under long-life service condition are investigated. The results show that the creep life of the alloy at 980 °C/90 MPa is measured to be 9714 h. During creep, the various morphologies of γ' and γ phases display in the different regions of sample. Wherein, the γ' phase in the region near fracture is firstly transformed into the rafted structure, while the γ' phase in the stress-free region exhibits the bunch-like structure. The size of the rafted γ' phase in thickness increases from 0.4 μm to 1.8 μm as the time of creep prolongs to 9714 h, the coarsening regularity of the rafted γ' phase in thickness obeys the parabolic law. Moreover, the parameters and misfits of γ'/γ phases in the alloy increase with the creep time. When the creeping time is less than 3000 h, the deformation mechanisms of alloy are dislocations slipping in the matrix channels and climbing over the rafted γ' phase. In the later stage of creep, the deformation mechanisms of alloy are dislocations slipping in the matrix channels and shearing into the rafted γ' phase, the alternate activation of the main/secondary slipping dislocations promotes the coarsening and twisting of the rafted γ'/γ' phases. Wherein, the coarsening of the rafted γ' phase is thought to be a main reason of the alloy having a better creep resistance and longer life.

1. Introduction

The creep behavior of directional solidified nickel-based superalloys is closely related to their microstructure and micro-deformation mechanism, especially, the combined action of temperature and stress fields results in the obvious change of microstructure. For example, the coarsening and rafting transformation of γ' phase occurs during primary creep at high temperature, which results in the formation of dislocation networks in the interfaces of γ'/γ phases [1–4]. Wherein, during creep of directional solidified alloy, the rafted γ' phase with various configurations is formed in the different columnar crystals, which has a significant effect on the deformation mechanism of alloy.

Although the creep behavior and deformation mechanism of nickel-based superalloys during creep has been extensively studied [5–8], the hot parties in the engines of military transport and civil aircrafts need the longer life, and the dependence of the creep behavior of the hot parties under the long-life service condition on the microstructure evolution and deformation mechanism is still unclear. Especially the creep behaviors of the blade parties under the long-life service condition are related to the security dependability of engineering applications, therefore, it is necessary to study the dependence of the creep behavior for the hot parts under the long-life service conditions on the microstructure evolution [9–11].

The lattice parameters and misfits of γ' and γ phases in alloy change with the chemical compositions [12,13], and the change of the ones in alloy occurs during aging with/free stress [14–16]. Namely, the various alloys or same alloy at different states display the various parameters and misfits of γ'/γ phases [17,18]. Because the combined action of the misfits stress in the γ'/γ phases and applied stress during creep is thought to be the main driving force of dislocation motion [19,20], the behavior and resistance of alloy during creep is related to the misfits of γ'/γ phases. Although the dependence of the misfits of γ'/γ phases on the creep behavior of nickel-based single crystal superalloys have been reported in some literatures [21–24], the dependences of the lattice parameters and misfits in directional solidified superalloys on the service time are unclear. Especially under long-life service conditions, the change

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regularity of the parameters and misfits, the evolution features of γ'/γ phases in the alloy is still unclear.

Thereafter, by means of measuring creep properties of a directional solidified nickel-based superalloy under long-life service conditions, combined with the microstructure observation under SEM, TEM and measurement of parameters and misfits, the dependence of microstructure evolution, misfits and deformation mechanism on creep time is investigated. This may provide a theoretical basis for the prediction-life of the alloy under long-life service conditions.

2. Experimental procedure

By means of vacuum directional solidification furnace with high temperature gradient, the bars of a directional solidified nickel-based superalloy with $\Phi 16 \times 140$ mm in size were prepared along the [001] orientation. The chemical composition of the nickel-based superalloy is given in Table 1. The used heat treatment regimes are given as follows:

After fully heat treatment, the bars of the alloy are machined into the cylindrical creep samples with 5 mm in diameter, and then the creep property of alloy at 980 °C/90 MPa is measured to plot the creep curve. Moreover, some other bars of the alloy are machined into the plate-like creep samples with the cross section of 4.5 mm × 2.5 mm and the gauge length of 20 mm. After mechanical grinding and polishing, the samples are placed in the creep testing machine with GWT504 model to apply the tensile loading of 90 MPa at 980 °C, and the creep tests are stopped after the samples are crept for 500 h, 1000 h, 2000 h and 3000 h, respectively. After the samples with different states are mechanically grinded and polished, the microstructure of the ones is observed, under Scanning electron microscopy (SEM) and Transmission electron microscope (TEM), to examine the evolution features of microstructure of the alloy crept for different times. The etching solution with composition as 10 g $(NH_4)_2SO_4$ + 10 g $(C_6H_8O_7 \cdot H_2O)$ + 1200 ml H_2O is used to conduct the electrolytic corrosion of the samples with various states.

After crept for 500 h, 1000 h, 2000 h, 3000 h and 9714 h up to fracture, respectively, under the applied stress of 90 MPa at 980 °C, the samples are cut into the plate-like specimen with 0.5 mm in thickness along the (100) plane. The specimens are grinded and polished to the films with the size of 50 μm in thickness, and then the films are thinned by twin jet polishing technique using an electrolyte consisting of 10% perchloric acid and 90% ethanol at 253 K, for preparing the TEM sample to observe the deformation feature of alloy under JEM-2000FXII transmission electron microscope.

After the samples are crept for 500 h, 1000 h, 2000 h, 3000 h and 9714 h up to fracture, respectively, under the applied stress of 90 MPa at 980 °C, the X-ray diffraction spectrums of the samples at room temperature are measured. Due to the close lattice parameters of γ and γ' phases, the diffraction peaks in XRD curves consist of the superposition of ones of γ' and γ phases. Therefore, according to the difference of γ'/γ volume fraction, the synthetic diffraction peaks of γ'/γ phases in the certain angle are chosen, and then separated by means of origin software. The 2θ angles in the separated diffraction peaks of γ'/γ phases are inserted into the formula (1) to obtain the spacing of the crystal planes, so that the parameters and misfits of γ and γ' phases in the alloy crept for different times are calculated, respectively, according to the formula (2). In the measurement of X-ray diffraction spectrums, the Cu target with wavelength of 0.15406 nm is used.

$$2d \sin\theta = \lambda \tag{1}$$

$$a = d\sqrt{h^2 + k^2 + l^2} \tag{2}$$

Table 1
Chemical compositions of the superalloy (mass fraction, %).

Cr	Co	W	Mo	Al	Ti	Ta	Hf	B	C	Ni
8.68	9.80	7.08	2.12	5.24	0.94	3.68	1.52	0.012	0.09	Bal.

3. Experimental results and analysis

3.1. Creep behaviors and deformation feature

After fully heat treatment, the obvious dendrite exists still in the alloy, as shown in Fig. 1(a), the growth direction of primary dendrite in alloy is marked by the vertical line in Fig. 1(a), the growth direction of secondary dendrites is marked by the horizontal line, and the growth directions of the primary and secondary dendrites are perpendicular each other. The amplification morphology of the sample is shown in Fig. 1(b), which indicates that, after solution treatment at high temperature, the boundaries between the grains are still kept in the alloy. Some fine carbide particles are distributed in the boundary regions, as marked by the short arrow in Fig. 1(b), and some thicker carbide bulk in alloy are decomposed for distributing within the grain in the form of the networks, as marked by the longer arrow. The cubical γ' phase about 0.4 μm in size is distributed in the inter-dendrite and dendrite region, the amplification morphology of the one is shown in the right-up of Fig. 1(b). Therefore, it is concluded from Fig. 1 that the microstructure of the alloy consists of γ matrix, γ' and carbide phases, and the cubical γ' phase is coherently embedded in the γ matrix.

The creep curve of the alloy at 980 °C/90 MPa is shown in Fig. 2, three stages of the primary, steady-stated and accelerated creep are included in the curve. The transient strain of the alloy occurs when the load is applied at high temperature, the primary creep stage of alloy lasts about 110 h. And then the creep of alloy enters the steady-state stage, the strain rate of the alloy during steady-state creep is measured to be 0.000106%/h. After crept for 7500 h, the creep of alloy is still in the steady-state stage, and then the creep of alloy enters the accelerated stage, the creep life of alloy is measured to be 9714 h. This indicates that the alloy displays a longer creep life under long-life service condition.

After crept for 500 h, 1000 h and 2000 h at 980 °C/90 MPa, the microstructure of the alloy is shown in Fig. 3. Wherein, the microstructure of alloy crept for 500 h is shown in Fig. 3(a), which indicates that the γ' phase in alloy has transformed into the rafted structure along the direction perpendicular to stress axis, the size of the rafted γ' phase in thickness is about 0.7 μm, and dislocations networks are distributed in the interfaces of the rafted γ'/γ phases. And it is thought that the deformation mechanisms of alloy during creep at the time being less than 500 h are only dislocation slipping in the γ matrix channel because no dislocation shearing into the rafted γ' phase is detected in the alloy. When the slipping of dislocations in γ matrix channels is hindered by the rafted γ' phase, the dislocations may react with the dislocation networks, in which the component of dislocation decomposition changes the direction of the original movement to promote the climbing of them. The magnified morphology of dislocation networks is shown in the right-up side of Fig. 3(a). The deformation feature of alloy crept for 1000 h is shown in Fig. 3(b), indicating that the sizes of the rafted γ' phase and matrix channels in thickness increases obviously. But no dislocations shearing into the rafted γ' phase is detected in the alloy still, and the regular dislocation networks are