



Elements distribution and deformation features of a 4.5% Re nickel-based single crystal superalloy during creep at high temperature

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ABSTRACT

By means of creep property measurement, microstructure observation and atom probe tomography, the elements distribution and deformation features of a 4.5% Re nickel-based single crystal superalloy during creep are investigated. Results show that after fully heat treated, the elements Al and Ta are mainly distributed in γ' phase of alloy. After the alloy is crept up to fracture at 1100 °C/137 MPa, the change of the elements distributing in γ and γ' phases occurs, wherein, the concentration decrement of the elements Al and Cr in γ' and γ phases is attributed to their oxidation which may consume the ones. Moreover, some of the atoms Re, W and Mo dissolving in γ' phase are repelled into γ matrix to reduce its internal energy. And the peak concentration of the atoms Re and W appears in the γ phase near the transition region, the atoms Re, W in the peak region cause the lattice distortion to increase the resistance of dislocations motion, which may improve the creep resistance of alloy. In the later stage of creep, dislocations shearing into the rafted γ' phase may cross-slip from {111} to {100} plane to form the configuration of K-W locks, which may restrain the slipping of dislocations to improve the creep resistance of alloy. Wherein, the atom Re with lower diffusion coefficient may delay the dislocations in K-W locks for slipping and cross-slipping, which is thought to be the main reason of the K-W locks keeping in 4.5%Re alloy during creep at 1100 °C.

1. Introduction

Nickel-based single crystal superalloys have been widely used to make the blade parts of the advanced aero-engine due to their excellent creep resistance at high temperature [1,2]. But with the increase of the aero-engines work efficiency, the mechanical and creep properties of alloys at high-temperature need to be improved. Adding refractory elements, such as W, Mo, Ta and Re, can efficiently enhance the temperature capability and creep resistance of alloy at high temperature [3,4], and adding 3 wt% and 6 wt% element Re is thought to be the composition feature of the second and third generations single crystal nickel-based superalloys [5].

Microstructure of single crystal nickel-based superalloys consists of cubical γ' phase embedded coherently in γ matrix. The reason of the Re-containing alloys displaying the excellent mechanical properties at high temperature is attributed to the low diffusion coefficient of Re and the Re atomic clusters distributing in γ matrix to hinder the dislocations motion [6,7]. The investigation on creep mechanisms indicates that, in the primary creep stage of alloys at intermediate temperature, the activated dislocations may overcome the Orowan resistance for slipping

on the {111} planes of γ matrix [8,9]. And two sets dislocations slipping on γ matrix may react to form the dislocation networks, which can promote the dislocations climbing over γ' phase during steady state creep to delay the stress concentration [10]. Moreover, during creep at 760 and 980 °C, the super-dislocations shearing into the γ' phase may cross-slip from {111} to {100} planes to form the K-W locks (Kear-Wiltsdorf locks) [11], which may restrain the slipping of dislocations on {111} plane to improve the creep resistance of alloys.

In the primary stage of creep at high temperature, the cubical γ' phase in alloy is transformed into the rafted structure along the direction perpendicular to the stress axis [12,13], and the dislocation networks are distributed in the interfaces of the rafted γ/γ' phases [14]. During steady state creep, the deformation mechanism of Re-containing superalloys is dislocations slipping in γ matrix and climbing over the rafted γ' phase [15]. However, the interfacial dislocations are damaged in the latter stage of creep, so that the dislocations piled up in γ matrix may shear into γ' phase along the damaged networks [16] due to the decrease of elastic energy [17]. And the plastic deformation of alloys increases with the creep, due to the alternant activation of primary/secondary slipping dislocations [16], which results in the cavities and

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micro-cracks appearing in the twisted interfaces of the rafted γ'/γ phases [17]. As the creep goes on, the propagation of the crack along the direction perpendicular to stress axis occurs, until the creep fracture of alloy [18], because the formation of the cavities and cracks may decrease the creep resistance of alloys.

Although the creep behaviors of the Re-containing nickel-based single crystal superalloys at high temperature had been reported [19,20], few literatures report the dependence of element re-distribution on the creep. Moreover, the deformed features of alloys during creep are related to their chemical compositions and service conditions [21], and it is unclear whether the K-W locks may be kept in the rafted γ' phase of alloy during creep at temperature near 1100 °C. Especially, whether the concentration distribution of elements in the rafted γ'/γ phases to be changed during creep is still an open question.

In this paper, by measuring creep properties and microstructure observation, combined to the composition analysis of alloy before/after creep, the elements distribution and deformation features of a 4.5% Re nickel-based single crystal superalloy during creep at high temperature are investigated.

2. Experimental Procedure

A 4.5% Re nickel-based single crystal superalloy with [001] orientation had been prepared by means of selecting crystal method in a vacuum directional solidification furnace under a high temperature gradient. The nominal composition of the alloy is Ni-6Al-6Cr-6Co-5W-3Mo-7Ta-4.5Re (wt%), the growth direction of the sample is determined to be within 7° deviating from [001] orientation by means of Laue-back reflection method. The heat treatment regime of the single crystal nickel-based superalloy is shown in Table 1.

After fully heat treatment, the bar of the single crystal alloy was machined into the tensile creep samples with cross-section of 2.5 mm × 4.5 mm and the gauge length of 20.0 mm along [001] orientation. After polished mechanically, the samples are placed in the creep machine with GWT504 model to measure the creep property at 1100 °C/137 MPa. After crept up to fracture, the microstructure of the sample is observed under the scanning electron microscope (SEM) and transmission electron microscope (TEM) to reveal the deformation and damage features of alloy during creep. Combined with the contrast analysis of dislocation configuration, the deformation mechanisms of the alloy during creep at 1100 °C is investigated.

In order to investigate the effect of creep on the concentration distribution of elements in γ'/γ phases, after fully heat treated and crept up to fracture, the bars of the single crystal alloy are cut into the needle-like samples with a cross-section of 0.3 mm × 0.3 mm and length of about 14 mm along the [001] direction. After electrolytic polished, the samples are put into the 3D atom probe with LEAP4000XHR model to measure the concentration distribution of the elements in γ'/γ phases, respectively. In the process of the measurement, the samples are cooled to about 20–80 K under the super high vacuum condition (UHV), which may diminish the thermal vibration of atoms. The samples are connected to the anode to make the atoms on the top to be activated, and the evaporation rate of 0.01 ions/pulse and energy of 40 pJ/pulse is obtained under a pulse repetition rate of 125 kHz. The 2 dimension coordinate of the flight ion on the top of sample is recorded by position sensitivity probe, and the vertical coordinate of the ions is defined by cumulating of the ones layer by layer, so that the distribution mapping of the atoms with various elements in 3D is obtained further [22]. The analysis of the data is performed by using IVAS 3.6.8 software.

After reconstructed by 3D atom probe tomography, the distribution maps of Al and Cr atoms in the alloy before/after crept are shown in Fig. 1, where the sizes of needle samples are labeled. Here, the enriched regions of the Al and Cr atoms are distributed in the upside and down side of Fig. 1, respectively. According to the distribution of Al atoms in Fig. 1, the concentration of 10 at.% Al is defined as the isosurface of the Al clusters, as shown in the dark color regions of Fig. 1. After the alloy is crept up to fracture at 1100 °C/137 MPa, significant amount of fine Al clusters are distributed in γ matrix, as marked by the arrow in Fig. 1(c), the fine Al clusters are formed during cooling after the sample is crept up to fracture. The size of the fine Al clusters is about 10 nm, the distance of the fine Al clusters in γ matrix to the isosurface of Al clusters is about 15 nm, as shown in Fig. 1(c) and (d).

3. Experimental Results and Analysis

3.1. Microstructure Evolution During Creep

The creep curve of the alloy at 1100 °C/137 MPa is shown in Fig. 2. The creep life of alloy is measured to be about 164 h, the strain rate of alloy during steady state creep is about $4.6 \times 10^{-4} \text{ h}^{-1}$, and the strain of the alloy crept up to fracture is measured to be about 25.4%.

The microstructures of alloy at various states are shown in Fig. 3. After fully heat treatment, the cubical phase is identified as the γ' ordered phase by means of selected area electron diffraction, as shown in Fig. 3(a), and the chemical compositions of the cubical γ' ordered phase are measured to be Al 10.7%, Ta 14.3%, W 3.1%, Cr 3.2%, Co 4.1%, Mo 0.7% and Re 2.1%, (mass fraction, %), namely, the elements Al and Ta are enriched in the cubical γ' phase. Therefore, the Al clusters in Fig. 1 are identified as the γ' ordered phase according to the analysis results stated above, and the fine Al clusters in γ matrix in Fig. 1(c) and (d) are identified as the fine γ' phase formed during cooling of sample. Therefore, it may be concluded that the microstructure of the alloy consists of the cubical γ' phase embedded coherent in γ matrix, and the cubical γ' phase is regularly arranged along $\langle 100 \rangle$ orientation. Moreover, the edge sizes of the cubical γ' phase and width of the γ matrix channel are measured to be about 0.38–0.42 μm and 50 nm, respectively.

The microstructure of alloy crept for 100 h at 1100 °C/137 MPa is shown in Fig. 3(b), which corresponds to the steady state stage of alloy during creep. Wherein, the γ' phase in alloy has transformed into the N-type rafted structure along the direction perpendicular to the stress axis, the direction of the applied stress is marked by the arrows. It is indicated that significant amount of dislocations distribute in the γ matrix channels, as shown in the region A, few dislocations are detected within the rafted γ' phase, the dislocation networks are distributed in the interfaces of the rafted γ'/γ phases, as shown in the square frame of Fig. 3(b). The fact of no dislocation shearing into the rafted γ' phase suggests that the deformation feature of alloy during steady state creep is dislocations slipping in γ matrix and climbing over the rafted γ' phase. Moreover, it is indicated according to the smaller spacing distance of networks that the alloy has a bigger lattice misfit [23,24],

It is thought by analysis that dislocations in γ matrix move to the interfaces of the rafted γ'/γ phases to react with the networks, which may change the original moving direction of dislocations to promote dislocations climbing over the γ' rafts [25]. Therefore, the dislocation networks at the interfaces of γ'/γ phases may relax the stress concentration to delay the dislocations shearing into the γ' phase.

Table 1
Used heat treatment regime of the 4.5%Re single crystal nickel-based superalloy.

Homogenous	Solution	First aging	Secondary aging
1280 °C × 2 h + 1310 °C × 8 h	1325 °C × 10 h	1150 °C × 4 h	870 °C × 24 h

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