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Dislocation motion during high-temperature low-stress creep in Ru-free and Ru-containing single-crystal superalloys

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ABSTRACT

Creep deformation of the two experimental single crystal superalloys at high-temperature low-stress (1140 °C/137 MPa) has been analyzed through transmission electron microscopy. Emphasis is placed on elucidating the dependence of dislocation motion on microstructural evolution. The detailed analysis demonstrated that the stacking fault energy of the γ matrix significantly decreased with the addition of Ruthenium (Ru). The stacking faults presenting in the γ matrix after heat treatment has been rarely reported previously. During the primary creep stage, the dislocations can easily cross-slip on the different {1 1 1} planes in the horizontal matrix and leave 60° dislocation loops on the (0 0 1) γ/γ' interfacial plane. Furthermore, calculations demonstrated that it is difficult for the slipping dislocations to bow into the vertical γ matrix channel. In the early stages of steady state creep, the interfacial dislocations reoriented slowly from the ⟨1 1 0⟩ slipping direction to the ⟨1 0 0⟩ well misfit stress relief direction. On the other hand, few dislocations shearing into the rafted γ' phase have been observed. In fact, during the middle stages of the steady state creep, although perfect dislocation networks have formed, some dislocations shearing into the γ' phase have also been observed. In addition, the a ⟨0 1 0⟩ type superdislocations (some with non-compact core) have also been observed in the two experimental alloys. At last, the Ru-containing alloy possesses more negative lattice misfit, denser γ/γ' interfacial dislocation networks and higher microstructural stability, thus can maintain a minimum creep rate in the steady state stage and have a longer creep life.

1. Introduction

Nickel-base single crystal superalloys have been widely used as important materials for turbine blades in modern aircraft because of their excellent mechanical properties at elevated temperatures [1–3]. These alloys have disordered solid solution gamma matrix (γ, fcc structure) and strengthened by high volume fraction (about 65%) of the ordered gamma-prime precipitates (γ', Ni_3Al) with $L1_2$ structure. The matrix and the precipitates have a cube/cube orientation relationship. Due to a small difference in the lattice parameters between the two phases, a high misfit stress is present in the superalloys [2]. The misfit stress and the applied stress are of vital importance in driving the motion of dislocations during high-temperature low-stress creep [4]. During creep, the dislocation motion includes dislocation climb, cross-slip, reaction and pairwise shearing into the cubic or rafted γ' precipitates [2]. The dislocation movements have been investigated extensively under low-temperature high-stress and high-temperature low-stress creep conditions [2,4–10]. However, the creep tests in the previous works usually focused in the temperature range of 750–1100 °C [4–9]. In order to improve the net thermal efficiency further, higher temperature capabilities are required for the superalloys. For this purpose more refractory alloy elements, such as Re and Ru, have been added [11–18]. In fact, a few studies on the creep behavior at very high temperature (above 1100 °C) have been reported [10,19], and no reports on the microstructural evolution of dislocation movement were found elsewhere. Consequently, two experimental alloys with different Ru-containing (named 0Ru alloy and 3Ru alloy) have been prepared to reveal the microstructural evolution during creep at very high temperature (1140 °C). In addition, the correlations between the microstructural evolution and creep curves have been analyzed in detail.

2. Experimental procedures

The nominal chemical compositions of the two experimental superalloys are listed in Table 1. Both alloys have similar chemical compositions except 3 wt.% Ru addition in the 3Ru alloy. The single crystal bars of the [0 0 1] orientation were cast by means of crystal

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selection method in a directional solidification vacuum furnace under a high thermal gradient. The solution temperature of the two experimental alloys was determined by both the metallurgical analysis and the differential thermal analysis (DTA). Since the incipient melting temperature of the two experimental alloys is similar, both alloys were subjected to the same heat treatment. A two-step solution heat treatment and a two-step aging heat treatment were carried out on each alloy as follows:

1315 °C, 16 h + 1325 °C, 16 h + AC (air cooling) → 1150 °C, 4 h + AC → 870 °C, 24 h + AC.

After heat treatment, the γ' precipitates regularly embedded in the γ matrix. For the two alloys, the volume fraction of the γ' precipitates is approximately 70% and average size along the cube edge is approximately 0.3 μm. The standard deviation of average size of the 0Ru and 3Ru alloys is about 0.070 and 0.066 μm, respectively. In addition, the mean width of the γ matrix is approximately 60 nm for the two experimental alloys. Cylindrical creep specimens, with a diameter of 5 mm and a gauge length of 25 mm, were machined from the post-heat treated single crystal bars.

Creep rupture tests for both alloys were conducted at 1140 °C/137 MPa. The results of the experiment aiming at establishing the dependence of dislocation motion on the microstructural evolution are reported here. In order to clearly show this relationship further, the specimens have been interrupted after 2, 15 and 45 h creep tests for each experimental alloy, respectively. These interrupted specimens were cooled under load to maintain the deformation microstructures. Foils for transmission electron microscopy (TEM) were cut perpendicular to the longitudinal axis from the gage section of the post-creep specimens. For the rupture samples, the thin foils were cut about 5 mm away from the fracture surface. These foils were mechanically thinned down to a thickness of approximately 50 μm. After grinding, TEM foils were electrochemically thinned using a twin jet polisher in a solution of 5% acetic acid, 10% perchloric acid and 85% ethanol by volume. The optimum jet polishing conditions were determined to be at a temperature of −25 °C and a current of 30 mA. The resulting foils were examined in a JEM 2100 transmission electron microscope operating at 200 kV.

3. Results and discussion

To study the evolution of microstructures and the process of dislocation motion, creep tests covering the temperature of 1140 °C/137 MPa were performed. In general the tests were not run to fracture, they were interrupted at primary stage, early steady state stages and middle steady state stages for the purpose of preserving the microstructure for microscopy.

3.1. Creep curves

Representative creep curves at 1140 °C/137 MPa for the two experimental alloys are shown in Fig. 1. The inset shows the detail of 15 h initial creep. No incubation period and instantaneous plastic straining upon the application of the stress for the two experimental alloys have been observed. As a matter of fact, the two alloys are in a steady state creep region after 2 h test. The creep rupture life is approximately 93 and 113 h for the 0Ru alloy and the 3Ru alloy, respectively. It can be seen that the 3Ru alloy has a higher creep strain than the 0Ru alloy in the initial 13 h. However, after the initial 13 h the creep strain of the 0Ru alloy gradually higher than that of the 3Ru alloy until rupture.

Table 1
Nominal chemical compositions (wt.%) of the experimental superalloys investigated.

Alloy	Co	Al	Cr + Mo + W + Ta	Re	Ru	Ni
0Ru	12	6	19.4	5.4	0	Bal.
3Ru	12	6	19.4	5.4	3	Bal.

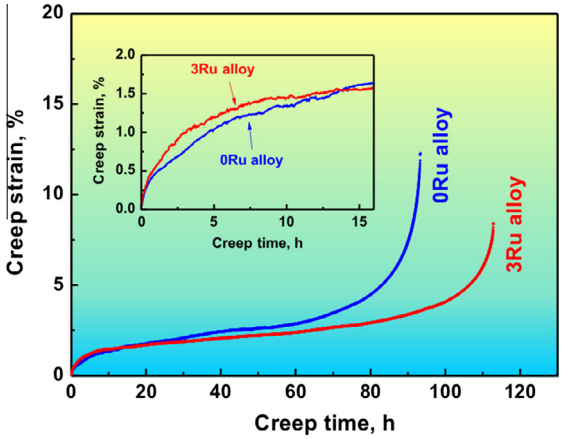

Fig. 1. Creep curves for the 0Ru alloy and 3Ru alloy at 1140 °C/137 MPa. The inset shows the detail of 15 h initial creep.

3.2. Dislocation configurations in the interrupted and the ruptured creep specimens

To study the effect of the dislocation motion on the creep process in single crystal superalloys, the microstructural evolution was monitored by observing the heat-treated, the interrupted and the ruptured creep specimens.

3.2.1. The heat treatment materials

Fig. 2 shows micrographs of the γ/γ' structure of the 0Ru alloy (Fig. 2a and b) and the 3Ru alloy (Fig. 2c and d) under the heat treated condition. Isolated misfit dislocations at the γ/γ' interface are occasionally observed, as shown in Fig. 2a and c. The micrographs in Fig. 2a and c are representative of the typical structure over most regions in the heat treated materials. On the other hand, regions of "grown in" dislocations in Fig. 2b are also occasionally seen in the 0Ru alloy. These grown-in dislocations have the same Burgers vector and all lie in the matrix channel. It is interesting that the stacking faults in the γ matrix of the 3Ru alloy are occasionally observed, as shown in Fig. 2d. The formation of the stacking faults during the heat treatment process is unusual, and is indicative of very low stacking fault energy. This phenomenon has been rarely reported previously. The driving force for the a/2 ⟨1 0 1⟩ perfect dislocations dissociating and extending must be related to the misfit stress. In previous study [20], Ru additions resulting in the lattice misfit more negative has been confirmed. Consequently, the misfit stress of the 3Ru alloy is higher than that of the 0Ru alloy. According to the above explanations, the stacking faults presenting in the γ matrix is reasonable. For the two experimental alloys, the γ' phase is free from dislocations.

3.2.2. After 2 h of creep

Fig. 3 shows the dislocation configuration of the 0Ru alloy interrupted after creep testing for 2 h at 1140 °C/137 MPa. Several kinds of typical slip dislocations with different characters have been observed as shown in Fig. 3a. These slipping dislocations are temporarily confined within the [0 0 1] γ matrix channel. Dislocation "AB", as shown in the bottom right corner of Fig. 3a, is cross gliding from the $(1\bar{1}1)$ plane to the (1 1 1) plane in the γ matrix channel. At "s", part of "AB" has bowed into the [0 1 0] vertical matrix channel. The stereoscopic configuration of dislocation "AB" is schematically illustrated in Fig. 3b. Geometrical considerations indicate that the segments of dislocation "AB" in the [0 0 1] γ matrix having special line directions (the [1 1 0] and $[\bar{1}10]$ directions) can only be of 60° dislocation [2,4,21]. Thus, the dislocation segments in the [0 1 0]