

# Creep properties and microstructure evolution of nickel-based single crystal superalloy at different conditions



Zhen-xue SHI, Jia-rong LI, Shi-zhong LIU, Xiao-guang WANG

Science and Technology on Advanced High Temperature Structural Materials Laboratory,  
Beijing Institute of Aeronautical Materials, Beijing 100095, China

Received 29 July 2013; accepted 17 December 2013

**Abstract:** The creep properties of nickel-based single crystal superalloy with [001] orientation was investigated at different test conditions. The microstructure evolution of  $\gamma'$  phase, TCP phase and dislocation characteristic after creep rupture was studied by SEM and TEM. The results show that the alloy has excellent creep properties. Two different types of creep behavior can be shown in the creep curves. The primary creep is characterized by the high amplitude at test conditions of (760 °C, 600 MPa) and (850 °C, 550 MPa) and the primary creep strain is limited at (980 °C, 250 MPa), (1100 °C, 140 MPa) and (1120 °C, 120 MPa). A little change of  $\gamma'$  precipitate morphology occurs at (760 °C, 600 MPa). The lateral merging of the  $\gamma'$  precipitate has already begun at (850 °C, 550 MPa). The  $\gamma$  phase is surrounded by the  $\gamma'$  phase at (980 °C, 250 MPa). The  $\gamma$  phase is no longer continuous tested at (1070 °C, 140 MPa). At (1100 °C, 120 MPa), the thickness of  $\gamma$  phase continues to increase. No TCP phase precipitates in the specimens at (760 °C, 600 MPa), (850 °C, 550 MPa) and (980 °C, 250 MPa). Needle shaped TCP phase precipitates in the specimens tested at (1070 °C, 140 MPa) and (1100 °C, 120 MPa). The dislocation shear mechanism including stacking fault formation is operative at lower temperature and high stress. The dislocation by-passing mechanism occurs to form networks at  $\gamma/\gamma'$  interface under the condition of high temperature and lower stress.

**Key words:** single crystal superalloy; creep properties; microstructure evolution;  $\gamma'$  phase; TCP phase

## 1 Introduction

Nickel based superalloy single crystals have superior mechanical properties at elevated temperatures, which makes them the most suitable materials for the manufacture of turbine blades in aero engines. The temperature capability of the turbine blade has increased significantly for the past several decades. Some of the advances have been achieved through improving the content of refractory alloying elements [1–4]. For example, the mass fraction of refractory elements in the single crystal superalloys from the first generation to the third generation, such as CMSX-2, CMSX-4 and CMSX-10, is 14.6%, 15.4% and 20.7%, respectively [5]. The Re content in the third generation single crystal superalloys CMSX-10 [1] and RenéN6 [2] is 5.4% and 6%, respectively, which is higher than 3% in the second generation single crystal superalloys CMSX-4 and RenéN5 [5], so the temperature capability of the CMSX-10 and RenéN6 alloys has been improved by

about 30 °C. However, the superalloys with high fraction refractory elements are susceptible to the precipitation of deleterious topologically close packed (TCP) phases [6,7], and the creep properties decrease as a result of the TCP formation [8,9]. The temperature and stress will influence the precipitation of TCP. Moreover, the turbine blades bear various work conditions from start to stable course and undergo the processes from medial temperature to high temperature. The single crystal superalloys with various compositions display different creep behavior [10]. Although a large amount of investigations have demonstrated the creep behavior of nickel based single crystal superalloys only at intermediate temperatures or at high temperature [10–13], few references report that the creep feature and deformation mechanisms of the alloys from medial temperature to high temperature. In this study, the creep properties of the single crystal superalloy with [001] orientation were investigated at different conditions. The microstructure evolution of  $\gamma'$  phase, TCP phase precipitation and dislocation characteristic after creep

rupture were studied by SEM and TEM. The purpose of the investigation is to promote the development of new generation single crystal superalloy with high creep properties.

## 2 Experimental

Pure raw materials were used in this experimental study. A Ni–Cr–Co–Mo–W–Ta–Nb–Re–Al system single crystal with [001] orientation was cast by means of crystal selection method in the directionally solidified furnace with the temperature gradient of 80 °C/cm. The nominal chemical compositions dimension of the alloy are listed in Table 1. The crystal orientations of the specimens were determined with Laue X-ray back reflection method, and the crystal orientation deviations of the specimens were maintained within 10° from the [001] orientation. The single crystal specimens received standard heat treatment comprising of solution treatment (1340 °C, 5 h, AC) and two-step aging treatment (1120 °C, 4 h, AC) + (870 °C, 24 h, AC). The standard cylindrical specimens for creep tests were machined after heat treatment, and the creep tests were conducted at (760 °C, 600 MPa), (850 °C, 550 MPa), (980 °C, 250 MPa), (1100 °C, 140 MPa) and (1120 °C, 120 MPa) in air using DST-5 testing machine with furnace attachment. The samples were etched with solution of 5 g CuSO<sub>4</sub> + 25 mL HCl + 20 mL H<sub>2</sub>O + 5 mL H<sub>2</sub>SO<sub>4</sub> which dissolved the  $\gamma'$  phase. Microstructures of the creep ruptured samples were examined by S4800 scanning electron microscope (SEM). Foils for transmission electron microscopy (TEM) analysis were obtained by cutting 0.2 mm-thick discs perpendicular to the tensile axis of the specimens using electric discharge machine. Thin foils were prepared by twin-jet thinning electrolytically in the solution of 10% perchloric acid and 90% ethanol (volume fraction) at –10 °C using liquid nitrogen.

**Table 1** Nominal chemical compositions of experimental alloy (mass fraction, %)

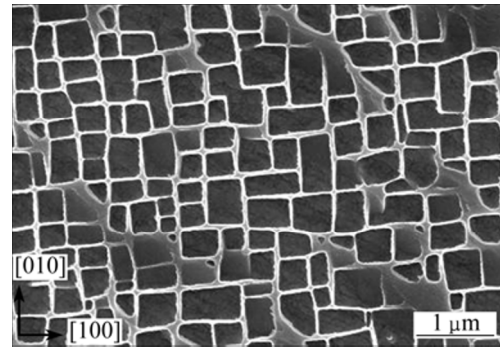
Cr	Co	Mo	W	Ta
2–4	7–10	2–5	6–9	7–10
Re	Nb	Al	Hf	Ni
3–5	0.5–1.5	5–6	0.1–0.5	Bal.

## 3 Results and discussion

### 3.1 Microstructure after heat treatment

Figure 1 shows the microstructure of the single crystal superalloy after standard heat treatment. It can be seen that the primary  $\gamma'$  and  $\gamma/\gamma'$  eutectic dissolve

completely after the high temperature solution treatment. The cubical  $\gamma'$  phase is regularly arranged along [100] direction. The average length of the  $\gamma'$  precipitates edge is about 0.45  $\mu\text{m}$ , the width of the  $\gamma$  matrix channel is about 0.05  $\mu\text{m}$ , and the volume fraction of the  $\gamma'$  phase in the alloy is more than 60%.



**Fig. 1** Microstructure of single crystal superalloy after standard heat treatment

### 3.2 Creep behavior

The creep curves of the nickel-based single crystal superalloy at different test conditions are shown in Fig. 2 and the corresponding creep properties are listed in Table 2. It can be seen that the alloy has long creep life at different test conditions, which indicates that the alloy has excellent creep properties. However, two different types of creep behavior can be shown by the creep curves. The first one is characterized by the high amplitude of primary creep and another is characterized by the limited primary creep strain. At (760 °C, 600 MPa) and (850 °C, 550 MPa), the primary creep strain which is about 4% and 3% accounts for 20% and 16% of total strain, respectively. The primary creep stage amplitude is dramatically smaller at (980 °C, 250 MPa), (1100 °C, 140 MPa) and (1120 °C, 120 MPa), less than 0.3%. The secondary creep stage is quite long and the main part of the creep life corresponds to the long secondary creep stage.

### 3.3 Microstructure of ruptured specimens under SEM

The microstructure on the longitudinal section in the ruptured specimens of the alloy at different conditions was observed by SEM. Figure 3 shows the microstructure apart from fracture surface 1 cm of the ruptured specimens. It can be seen from the observation that a little change of  $\gamma'$  precipitate morphology occurs at the condition of (760 °C, 600 MPa) (Fig. 3(a)). The vertical  $\gamma$  matrix becomes thinner and horizontal  $\gamma$  matrix becomes thicker slightly. No appreciable thickening of  $\gamma'$  precipitates seems to have taken place. The lateral merging of the  $\gamma'$  precipitates has already begun along the

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