Materials Science & Engineering A 580 (2013) 21–35

Contents lists available at SciVerse ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Effect of Ru additions on very high temperature creep properties of a single crystal Ni-based superalloy

X.P. Tan [a,b,c,*], J.L. Liu [a], T. Jin [a], Z.Q. Hu [a], H.U. Hong [d], B.G. Choi [b], I.S. Kim [b], C.Y. Jo [b], D. Mangelinck [c]

[a] Superalloy Division, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China
[b] High Temperature Materials Research Group, Korea Institute of Materials Science, 531 Changwondaero, Changwon, Gyeongnam 641-831, Republic of Korea
[c] IM2NP, UMR 7334, CNRS, Université Aix-Marseille, Av Escadrille Normandie-Niemen, Case 142, Marseille, Cedex 20 13397, France
[d] Department of Materials Science and Engineering, Changwon National University Sarim-dong, Uichang-gu, Changwon, Gyeongnam 641-773, Republic of Korea

ARTICLE INFO

Article history:
Received 7 April 2013
Received in revised form
9 May 2013
Accepted 10 May 2013
Available online 20 May 2013

Keywords:
Ni-based superalloy
Ru
Creep deformation
Interfacial dislocation
γ' rafting

ABSTRACT

The creep deformation of three single crystal Ni-based superalloys with various Ru contents has been investigated to clarify the effect of Ru additions at very high temperatures and low stresses conditions. The creep properties were significantly improved by Ru additions under the conditions of 1150 °C/100 MPa and 1180 °C/70 MPa. It is noted that obvious γ' phase dissolution occurs during the entire creep deformation at very high temperatures. It differs from the typical high-temperature creep curves that a short incubation period occurs before the primary creep stage during the creep deformation at very high temperatures. Ru additions are able to produce denser interfacial dislocation networks and improve the stability and homogeneous configuration of interfacial dislocations; and promote the high-temperature stability of γ' phase and formation of perfect γ' rafting. It is thus shown that Ru additions reduce the minimum creep rate, and prolong the secondary creep stage accordingly. The topological inversion of γ/γ' microstructure did not occur under both conditions. The origin of the rapid increase of creep rate is closely relevant to the unstable propagation of micro-cracks in the vicinity of porosity in necked regions.

1. Introduction

Single crystal Ni-based superalloys are the primary materials for the high pressure turbine blades and vanes in the advanced aircraft engines. Of great challenge is the temperature capability of single crystal Ni-based superalloys, since the turbine entry temperature (TET) has been increasingly raised in order to further improve the thermal efficiency and reduce the CO_2 emission in gas turbine systems [1,2]. The high pressure turbine blades experience the extremely complex and harsh service environment with centrifugal forces and thermal stresses behind the combustor. The high temperatures and stresses acting on the turbine blades would result in the occurrence of creep deformation and reduce their service life [2,3]. Therefore, the creep resistance is the most important mechanical properties of single crystal Ni-based superalloys. At present, the most advanced applicable single crystal Ni-based superalloy is able to serve at up to approximately 1100 °C with some strength and microstructural stability for a long term [4]. In general, the elevated temperature in excess of 1150 °C could be termed as the very high temperature for single crystal Ni-based superalloys. Under some emergency situations (such as one engine inoperative (OEI) rating during in-service operation of two engine helicopters), however, the short-time service temperature of single crystal Ni-based superalloys can reach up to 1200 °C [5,6]. Besides, one major aim of designing new generations of single crystal Ni-based superalloys is to increasingly enhance their creep resistance at elevated temperatures. Thus, it is necessary to carry out the related studies on the creep properties of single crystal Ni-based superalloys under extreme conditions of very high temperatures. In the past decades, the creep properties of various single crystal Ni-based superalloys have been widely studied [7–12]. However, there are few studies on the very high temperature creep properties of superalloys. Some studies on very high temperature non-thermal creep properties of single crystal Ni-based superalloy MC2 were carried out in order to simulate the short-time overheating service conditions [5,6,13–15]. With respect to the very high temperature isothermal creep properties, Reed et al. [16,17] investigated the creep damage mechanism of single crystal Ni-based superalloys under the very high temperature of 1150 °C. Caron et al. [18] analyzed the superior creep properties of single crystal Ni-based superalloy MC-NG at the very high temperature of 1150 °C.

* Corresponding author at: Universite Aix-Marseille, CNRS, Av Escadrille Normandie-Niemen, IM2NP, UMR 7334, Case 142, Marseille, Cedex 20 13397, France. Tel.: +33 4912 82710.
E-mail address: xptan1985@gmail.com (X.P. Tan).

0921-5093/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.msea.2013.05.028

In the recent years, Ru has become the symbol element in the new generations of single crystal Ni-based superalloys. It has been well accepted that the key roles of Ru are to improve the microstructural stability and creep resistance [18–21]. Thereinto, the ultimate purpose with Ru additions is to further enhance the creep properties. Previously, several papers were involved in the influence of Ru additions on the creep properties of single crystal Ni-based superalloys under various temperatures [21–25]. To the authors' knowledge, however, the relevant effects of Ru on the very high temperature isothermal creep properties of single crystal Ni-based superalloys have not been reported. In an attempt to elucidate the effect of Ru additions on the very high temperature creep properties of single crystal Ni-based superalloy, three alloys with various Ru contents (0 wt%, 2 wt% and 4 wt%) were employed to carry out the isothermal creep tests under the conditions of 1150 °C/100 MPa and 1180 °C/70 MPa. This paper aims at better understanding the so called Ru-effect mechanisms under the very high temperature conditions, and providing experimental evidence for the development of new generations of single crystal Ni-based superalloys with higher temperature capability.

2. Experimental

2.1. Materials

Three single crystal Ni-based superalloys with and without Ru additions were designed to study the effect of Ru additions on the very high temperature creep properties. The nominal chemical compositions of the alloys are listed in Table 1. According to their various Ru contents, these three alloys are named as 0Ru, 2Ru and 4Ru, respectively. The master alloys were melted by vacuum induction technique, and then directionally solidified into cylindrical bars (16 mm in diameter and 220 mm long) in an investment casting cluster mold in a Bridgman furnace with a withdrawal rate of 6 mm/min. Conventional helical starters were utilized to initiate single crystal growth. Only the single crystal bars deviating from ⟨0 0 1⟩ within 15° by Electron backscatter diffraction (EBSD) method were adopted. The heat treatment schemes for the three alloys are given in Table 2 [26].

2.2. Specimens preparation

The creep testpieces of diameter 6.0 mm and gauge length 25 mm were machined from the heat-treated single crystal bars.

Table 1
Nominal composition of the three experimental alloys (wt%).

Alloy	Ni	Al	Ti	Ta	Cr	Mo	W	Re	Ru
0Ru	Bal.	6	0.5	5	4	1	5	4	0
2Ru	Bal.	6	0.5	5	4	1	5	4	2
4Ru	Bal.	6	0.5	5	4	1	5	4	4

Table 2
Full heat treatment scheme of the three experimental alloys.

Alloy	Solid solution	Primary aging	Secondary aging
0Ru	(1318–1348) °C/10 h +1348 °C/15 h, AC*	1140 °C/4 h, AC	870 °C/24h , AC
2Ru	(1320–1350) °C/10 h +1350 °C/15 h, AC	1140 °C/4 h, AC	870 °C/24 h, AC
4Ru	(1310–1340) °C/10 h +1340 °C/15 h, AC	1140 °C/4 h, AC	870 °C/24 h, AC

* AC denotes the air cooling.

Tensile creep testing was performed at image processing displacement analyzer (IPDA) system, which consists of ATS 2410 series creep tester, high temperature furnace and image processing analyzer. The strain measurement is obtained from the visual sensing system in-situ monitoring the gauge mark of specimen. Testpieces were heated by the resistance furnace from Toshin Kogyo company (Tokyo, Japan) with working temperature range at 900–1500 °C during creep tests. When the targeting test temperature was achieved, soaking for 0.5 h will be performed. The creep strains start to be recorded once finishing soaking and tight clamping creep testpieces. Testpieces were creep ruptured at 1150 °C/100 MPa and 1180 °C/70 MPa and some others were interrupted at various amounts of accumulated creep strains.

An attempt was made to obtain the initial microstructures of the three experimental alloys during creep, the small pieces of heat-treated samples were heat treated followed by the simulative heating process of creep tests at 1150 °C and 1180 °C. Namely, (25–970) °C/2 h+970 °C/10 min+(970–1150) °C/1.5 h+1150 °C/0.5 h, water quenching (WQ) and (25–970) °C/2 h+970 °C/10 min+(970–1180) °C/1.5 h+1180 °C/0.5 h, WQ, respectively. Small pieces of solution heat-treated samples of the three alloys were isothermally exposed at 1140 °C for 8 h, 16 h, 27 h and 45 h, respectively, to observe the influence of Ru additions on γ' phase coarsening. In order to facilitate the γ' area fraction evaluation, measurements were made on the pre-rafted specimens of interrupted creep tests at 1150 °C/100 MPa after 7 h. Samples were taken from the specimen gauge, and then heat treated for 1 h at 1050 °C, 1150 °C, 1200 °C, 1250 °C and 1300 °C with WQ to freeze the high temperature microstructure. Samples for scanning electron microscopy (SEM) observation were prepared with sections parallel to the ⟨0 0 1⟩ tensile axis to obtain side view of the γ/γ' rafted microstructure. Based on the assumption that γ' rafts were greatly longer as compared to their thickness, so the area fraction of γ' phase approximately equals to its volume fraction [27].

The analysis of γ' phase dissolution in heat-treated microstructure was conducted on NETZSCH DSC 404C high-temperature-type differential scanning calorimeter (DSC). The cylindrical DSC samples (Φ3 × 1–2 mm) with approximate 200 mg were prepared. The heating and cooling rates are both 10 °C/min. A JEM-5800 SEM was used to examine the microstructure after heat treatment and creep tests. After creep tests, the specimens were cut into Φ3 mm discs of 500 μm in thickness normal to the [0 0 1] direction about 3 mm away from the fracture surfaces or necked regions and thinned down to 50 μm mechanically. They were then electrochemically polished by the twin-jet method, in a solution of 20% perchloric acid and 80% methanol at −30 °C and 30 mA. The field emission type JEOL JEM-2100F (200 kV) scanning transmission electron microscope (STEM) equipped with a GIF (Gatan Imaging Filter) was used to observe dislocations configuration.

3. Results

3.1. Heat-treated microstructure

Fig. 1 shows the microstructures of three alloys after full heat treatment. The average γ' size, γ' volume fraction and γ channel width are 0.46 μm, 64.3% and 58.7 nm for alloy 0Ru, 0.38 μm, 61.3% and 50.0 nm for alloy 2Ru and 0.29 μm, 56.7% and 42.0 nm for alloy 4Ru, respectively. As measured by the X-ray diffraction technique, the average γ/γ' lattice misfits of alloy 0Ru, 2Ru and 4Ru are +0.08%, −0.14% and −0.17% at room temperature, respectively. The detailed measurements can be seen in [26]. The change in γ/γ' lattice misfit was caused by the variation of partitioning ratios of alloying elements via Ru additions [26].