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## MATERIALS SCIENCE & ENGINEERING

Materials Science & Engineering A

# Effect of multiple alloying additions on microstructural features and creep performance at 950 °C and 400 MPa in Ru-containing single crystal superalloys



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#### ARTICLE INFO

Keywords: Microstructure Creep Lattice misfit Ru Superalloys

#### ABSTRACT

Microstructural features, including y channel width, y' size, y' volume fraction, y-y' lattice misfit, TCP phase as well as dislocation substructures had influence on the creep performance in Ni-base single crystal superalloys. However, relatively limited work has been conducted to investigate the effect of microstructural features based on various alloving additions on creep properties in Ru-containing single crystal superalloys. In this study, the creep tests were conducted at 950 °C and 400 MPa in experimental alloys with different levels Co (7.0 wt% and 15.0 wt%), Cr (3.5 wt% and 6.0 wt%), Mo (1.0 wt% and 2.5 wt%) and Ru (2.5 wt% and 4.0 wt%) additions, and the microstructures during creep were characterized in detail. Co and Ru were found to decrease the y channel width, respectively. The high level of Cr addition decreased the y' volume fraction and promote the TCP phase formation during creep. The addition of Mo decreased the y channel width and also acted as a TCP former. The y-y' lattice misfit was increased to more negative by the individual additions of Co, Ru, Cr, and Mo, respectively. The joint addition of microstructural stabilizers Co and Ru inhibited the TCP phase formation during creep. The precipitation of TCP phases served as one of the main factor to decrease the creep property in alloys with high level of Cr and Mo additions, respectively. The synergistic effect of Mo and Ru additions in the alloy with high level of Co content was found to increase the y-y' lattice misfit and the amount of stacking faults in y matrix during creep process, which improved the creep resistance at 950 °C and 400 MPa. This study is helpful to understand the effect of alloying elements additions and microstructures on creep performance and to get better improvement of physical metallurgy knowledge and alloy design in Ru-containing single crystal superalloys.

#### 1. Introduction

Ni-base single crystal superalloys are widely used as turbine blades in aircraft engines and serviced for thousands of hours under sufficient high temperature and pressure, because of their excellent high temperature mechanical properties. Generally, the microstructural degeneration of superalloys during creep procedure are mainly attributable to the directional coarsening and rafting of  $\gamma'$  precipitates, TCP phase precipitation and dislocation movement. Thus, to achieve the excellent creep resistance, it is necessary to investigate the microstructural features, including  $\gamma$  channel width,  $\gamma'$  size and volume fraction,  $\gamma$ - $\gamma'$  lattice misfit, TCP phase as well as dislocation substructure during creep, meanwhile, effects of alloying additions should be taken into consideration. During creep procedure at high temperature, the creep strength of Ni-base superalloys results from hardening by ordered  $\gamma'$  precipitates (Ni<sub>3</sub>Al) with L1<sub>2</sub> structure [1,2]. The single crystal superalloys with higher volume fraction of  $\gamma'$  precipitates exhibited better high temperature creep property, while the excess value of  $\gamma'$  volume fraction led to the decrease of creep performance [1,3–5]. Furthermore, the lattice misfit of  $\gamma$ - $\gamma'$  phases had important influence on the microstructures and creep properties in Ni-base single crystal superalloys. The effects of the symbol and scale of  $\gamma$ - $\gamma'$  lattice misfit have been investigated, and the negative  $\gamma$ - $\gamma'$  lattice misfit lead to the cuboidal  $\gamma'$  precipitates. Previous research showed that increasing the lattice misfit of  $\gamma$ - $\gamma'$  phases raised the rafting tendency of  $\gamma'$  phases [6] and reduced the spacing of dislocation networks, which improved the creep resistance of preventing dislocation from cutting  $\gamma'$  phases at high temperature

http://dx.doi.org/10.1016/j.msea.2017.03.081 Received 8 February 2017; Accepted 22 March 2017 Available online 23 March 2017 0921-5093/ © 2017 Elsevier B.V. All rights reserved.

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[7-9]. However, the formation of  $\gamma'$  rafting structure with more negative lattice misfit had negative effect of creep performance at lower temperature and high stress [10–12].

Actually, the microstructural features and evolution during creep are determined by the composition of alloying additions. Cr was found to improve high temperature strength by increasing the y' volume fraction and y-y' lattice misfit in Ru-free and Rucontaining superalloys [9,13]. Mo was regarded as a potent solid solution strengthener which was found to increase the lattice misfit of  $\gamma$ - $\gamma'$  phases and elevate the creep property at high temperature [14–16]. However, the excessive additions of Cr and Mo destroyed the creep property by precipitating TCP phases [17]. In addition, Co has also been reported to elevate microstructural stability in single crystal superalloys [18-20]. Ru was added to enhance the microstructural stability and creep properties in modern Ni-base single crystal superalloys [9,19], but the cost was obviously increased by Re and Ru additions. Thus, it is of great interests to develop a series alloy that can meet the requirements of high temperature microstructural stability and creep performance, while a relatively lower cost than the typical 4th generation single crystal superalloys. Furthermore, the synergistic effect of microstructural stability elements (Co and Ru) and creep strengthening elements (Cr and Mo) on microstructural features and creep performance are still required to investigate. Although the effects of microstructure features on the creep properties at different temperature and stress have been studied for many years, the various alloying additions and their synergistic effects were rarely involved [12], especially in Ru-containing single crystal superalloys.

In this paper, microstructural features, including y channel width, size and volume fraction of  $\gamma'$  phase,  $\gamma$ - $\gamma'$  lattice misfit, TCP phase and dislocation substructure during creep process at 950 °C and 400 MPa were investigated quantitatively and systematically in Ru-containing single crystal superalloys with various alloving additions. The relationship among composition, microstructural parameters and creep properties were evaluated with emphasis on the role of Co, Cr, Mo and Ru additions. The objective of this paper is to elucidate the effects of alloying additions and microstructures on creep properties, and to provide the reference to the design and optimization of Ru-containing single crystal superalloys with high performance at low cost.

#### 2. Experimental

To better understand the influence of Co, Cr, Mo and Ru on creep properties, the measured chemical compositions of experimental alloys are listed in Table 1. These alloys were named as SC plus four numbers, which in turn represented the level of Co, Cr, Mo and Ru additions ("1" and "2" denoting the low and high level of individual elements, respectively). Co, Cr, Mo and Ru were assigned in two levels of contents: Co: 7.0 wt% and 15.0 wt%; Cr: 3.5 wt% and 6.0 wt%; Mo: 1.0 wt% and 2.5 wt%; Ru: 2.5 wt% and 4.0 wt%.

These experimental alloys were directionally solidified as single crystal bars with the growth orientation < 001 > (15 mm in diameter)and 150 mm in lengths) at the Beijing Institute of Aeronautical

Measured compositions of	the experimental	alloys	(wt%/at%)
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Materials. The cast experimental alloys were solution heat treated at high temperature in the range of 1300 °C-1335 °C for 8-16 h, and then aged at 1150 °C for 4 h and at 870 °C for 24 h. The creep rupture tests were conducted at 950 °C and 400 MPa after the standard heat treatment. In order to investigate microstructural evolution during creep, the creep testing was interrupted with 1.0% creep strain at 950 °C and 400 MPa. The samples were conducted by furnace cooling and air cooling after the creep rupture and interrupted tests, respectively. The lattice misfit of  $\gamma$  and  $\gamma'$  phases was determined by in-situ synchrotron X-ray diffraction (XRD) at room temperature. The singlecrystal plate parallel to the (001) plane after the standard heat treatment was utilized at beamline BL14B1 in the Shanghai Synchrotron Radiation Facility (SSRF) with a beam energy of E=18 keV ( $\lambda$ =0.6887 nm). The (004) diffraction pattern was recorded and analyzed with the Jandel Scientific PeakFit computer program.

Metallographic specimens were etched in a solution of 33% (volume fraction) acetic acid, 33% nitric acid, 33% deionized water and 1% hydrofluoric acid. And the microstructure was investigated using a ZEISS SUPRA 55 field-emission scanning electron microscope (FE-SEM) equipped with an energy-dispersive X-ray spectroscope (EDS). A back-scattered electron (BSE) detector was used to differentiate between the dendrite core and the interdendritic region. The volume fraction of  $\gamma'$  precipitates in the dendrite core was determined using the method recommended in previous studies [21] and analyzed by Photoshop software. The  $\gamma'$  size and  $\gamma$  channel width were measured using the Image-Pro software. At least five images were used for determining the size and volume fraction of y' precipitates in each alloy.

In order to identify the typical dislocation microstructure of different alloys during creep test, thin foils for transmission electron microscopy (TEM) observations were prepared by twin-jet thinning technique in a solution of 10% perchloric acid and 90% alcohol at -30 °C and 30 V. The microstructural observation and selective area diffraction pattern (SADP) for thin foils were conducted using a F30 transmission electron microscope operated at 300 kV.

#### 3. Results

#### 3.1. Initial $\gamma$ - $\gamma'$ microstructure

The initial  $\gamma$ - $\gamma'$  microstructures in the dendrite core of experimental alloys after the standard heat treatment were examined by SEM, the sizes and the volume fractions of y' precipitates as well as the y channel widths were measured. Fig. 1 presents the typical microstructure in the dendritic core of alloy SC1112 after the standard heat treatment, and the morphology of y' precipitates was cuboidal. In addition, it is worth to mention that all the experimental alloys had cuboidal y' precipitates in the dendritic cores and interdendritic regions aligned along {001} directions after the standard heat treatment. The y channel widths, sizes and volume fractions of  $\gamma'$  precipitates for the experimental alloys are listed in Table 2. The initial  $\gamma'$  sizes and  $\gamma$  channel widths were measured in the dendrite core for each alloy after the standard heat treatment and the average  $\gamma'$  size of the experimental alloys was close

Alloy	Ni	Al	Та	W	Re	Hf	Co	Cr	Мо	Ru
SC1111	Bal.	6.0/13.9	7.9/2.7	5.4/1.9	4.4/1.5	0.1/0.04	7.1/7.6	3.6/4.4	1.0/0.7	2.6/1.6
SC2111	Bal.	6.1/14.1	8.1/2.8	5.5/1.9	4.2/1.4	0.1/0.04	15.0/15.9	3.5/4.2	1.0/0.7	2.7/1.7
SC1211	Bal.	6.0/13.7	7.9/2.7	5.4/1.8	4.0/1.3	0.1/0.03	6.9/7.2	6.5/7.7	1.0/0.6	2.6/1.6
SC1121	Bal.	6.0/13.9	8.0/2.8	5.5/1.9	4.1/1.4	0.1/0.04	6.9/7.4	3.5/4.2	2.4/1.6	2.7/1.7
SC1112	Bal.	6.0/14.1	8.0/2.8	5.4/1.9	4.0/1.4	0.1/0.04	6.9/7.4	3.5/4.3	1.0/0.7	4.0/2.5
SC2212	Bal.	6.0/13.9	7.9/2.7	5.3/1.8	3.9/1.3	0.1/0.03	14.4/15.2	6.1/7.3	1.0/0.6	3.7/2.3
SC2122	Bal.	6.1/14.2	8.0/2.8	5.4/1.9	4.0/1.3	0.1/0.04	14.5/15.5	3.5/4.2	2.4/1.5	3.9/2.4

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