

# Effect of heat-treatment on microstructure and high-temperature deformation behavior of a low rhenium-containing single crystal nickel-based superalloy

Nairong Sun<sup>a,b</sup>, Lanting Zhang<sup>a,b,c,\*</sup>, Zhigang Li<sup>a,b</sup>, Aidang Shan<sup>a,b,c</sup>

<sup>a</sup> School of Materials Science and Engineering, Shanghai Jiao Tong University, 800 Dong Chuan Road, Shanghai 200240, PR China

<sup>b</sup> Shanghai Key Laboratory of Advanced High Temperature Materials and Precision Forming, Shanghai Jiao Tong University, 800 Dong Chuan Road, Shanghai 200240, PR China

<sup>c</sup> Gas Turbine Research Institute, Shanghai Jiao Tong University, 800 Dong Chuan Road, Shanghai 200240, PR China

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## ABSTRACT

A low rhenium-containing [001] oriented single crystal nickel-based superalloy with different  $\gamma'$  morphologies induced by various aging treatments was compressed from room temperature to 1000 °C. All the single crystal samples with different  $\gamma'$  morphologies exhibit anomalous yield behavior. The sample first aged at 1180 °C has the widest anomalous temperature domain and highest yield strengths. The sample first aged at 1000 °C has the highest anomalous peak stress temperature.

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## 1. Introduction

Nickel-based superalloys have been designed and widely used in the turbine industry and power plant for their superior strength at high temperatures. The superior strength of this kind of single crystal superalloy (SC) is attributed to its high volume fraction of regularly aligned cubical  $\gamma'$  precipitates which are coherent with  $\gamma$  matrix [1,2]. These  $\gamma'$  phases ( $L1_2$  structures) are ordered  $Ni_3Al$  phases whose volume percentage can be up to 70% for most advanced superalloys. To improve the high temperature strength, new generation SCs were developed by adding high content rare elements like Re and Ru [3–5]. The costly rhenium could result in the difficulty of solution heat-treatment and forming topologically close-packed phases (TCP) which are harmful to high temperature properties and limits the application of these superalloys [6]. So optimizing the microstructures of the available low-Re containing single crystal superalloys become an important potential way for better properties at low cost.

$L1_2$  phase embedded in the  $\gamma$  matrix is known to be the origin of unusual anomalous high temperature mechanical properties of superalloys. The abnormal temperature dependence of the flow stress has been attributed to the actuations of dislocations dissociated on various planes of precipitate particles in single crystal superalloys [1]. The

strength of a given superalloy depends on such factors as particle size, composition, volume fraction of  $\gamma'$  precipitates, etc. Thus, extensive investigations have been made on the effects of those factors on high temperature mechanical properties [7–11]. In this paper, a 2nd generation low Re-containing single crystal nickel-based superalloy, known as DD6, with different aging heat treatments were studied [12]. Few researches have been made on the high temperature properties of this single crystal superalloy [13,14], but no research has been made on the microstructure dependent compressive properties of this single crystal superalloy at high temperatures. Both the compositional and the structure effects of  $\gamma'$  on the compressive strength were investigated. The overall objective of this paper is to determine the roles of these factors on compressive deformation behavior and optimizing the microstructures for better mechanical properties of this low Re containing superalloy.

## 2. Experimental procedure

The nominal composition of DD6 single crystal investigated here is listed in Table 1. This is a Re-containing 2nd generation single crystal superalloy, which contains lower rhenium compared to other 2nd generation alloys such as CMSX-4 and René N5.

The single crystal bars were directionally solidified by the withdrawal process using a helical selector. All the cast rods tested in this experiment had deviations within 7° from the perfect [001] direction.

\* Corresponding author.

E-mail addresses: [snrf22@hotmail.com](mailto:snrf22@hotmail.com) (N. Sun), [lantingzh@sjtu.edu.cn](mailto:lantingzh@sjtu.edu.cn) (L. Zhang).

The as-casted rods were fully solution heat-treated at (1290 °C, 1 h)+(1303 °C, 2 h)+(1312 °C, 2 h)+(1320 °C, 15 h). After solution treatment, various multi-step aging heat treatments were performed to obtain various  $\gamma'$  sizes and morphologies. Only the first aging treatments were different for various multi-step aging treatments. In order to obtain close volume fractions of  $\gamma'$  phase for the samples, the second aging treatments were kept the same for them (Table 2).

The fully heat-treated samples were polished and etched by 20 g CuSO<sub>4</sub>+50 ml hydrochloric acid+100 ml distilled water for scanning electron microscopy (JEOL JSM-7600F).

The compressive tests were carried out from room temperature to 1000 °C for the 3 differently aging treated test cylinders along the [001] direction using a Gleeble 3500 integrated digital thermal and mechanical testing system. A strain rate of  $10^{-4} \text{ s}^{-1}$  was used in this study. The SC cylindrical specimen with a gauge length of 11 mm and a diameter of 7 mm was sectioned from the single crystal bars. Thin

foils perpendicular to the load axis were incised from some of the specimens. Discs were mechanically thinned to 0.1 mm and then electro-polished at  $-30 \text{ }^\circ\text{C}$  in a 5% perchloric acid+ethyl alcohol solution. The foils were observed using transmission electron microscopes operating at 200 kV (JEOL 2100F with EDAX).

### 3. Experimental results

The typical  $\gamma/\gamma'$  microstructures of these SC superalloys used various first aging heat-treatment rules are displayed in Fig. 1 and listed in Table 2. It can be followed by Fig. 1 and Table 2 that the average size of  $\gamma'$  particles precipitated in the N-1 SC sample is smallest and irregular compared to those of the N-2 and N-3 samples. The transition of microstructure for the  $\gamma'$  phase from irregular fine odd-shape to a regular cubical and larger figure owes to the coarsening of  $\gamma'$  phase

#### 3.1. Temperature dependence of the flow stress

Some examples of stress–strain curves are shown on Fig. 2 for the three differently aged single crystals. Each of the high temperature flow stress curve has a top value (arrowed in Fig. 2(a)) after yield and then falls down. For comparison, the 0.2% yield stresses for each single crystal samples are plotted as a function of temperatures in Fig. 3. All the  $\sigma_{0.2}$  stresses of the three samples exhibit obvious anomalous temperature dependences. For N-3 sample, the 0.2% yield stress increases with the ascending temperature and achieves a peak value of about 925 MPa. Its peak stress presents at around 800 °C, then it decreases rapidly. The yield flow stresses of N-2 and N-1 single crystal show normal negative temperature dependences before 600 °C compared to that of N-3 SC sample. They decrease from room temperature to 600 °C. After 600 °C, the flow stresses of N-1 and N-2 increase subsequently with ascending temperature, then arrive their peaks before sharp descending. The

**Table 1**

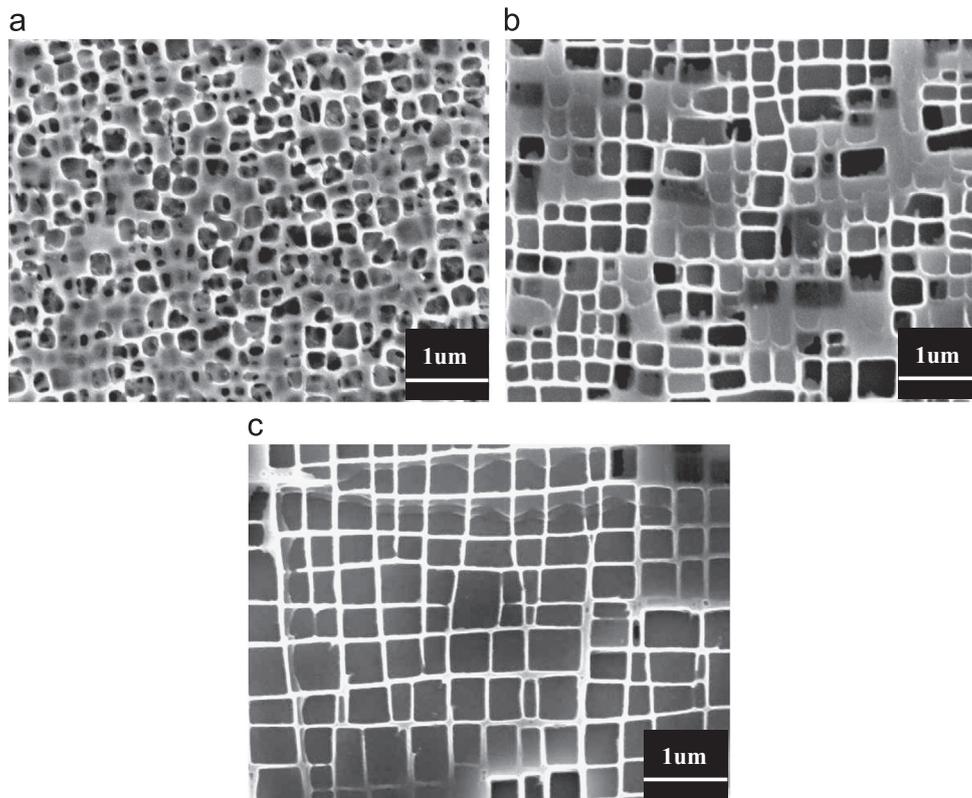
The nominal composition of DD6 single crystal superalloy (wt%).

Ni	Co	Mo	W	Al	Cr	Ta	Re	Hf
Bal.	9.0	2.0	8.0	5.7	4.3	7.5	2.3	0.1

**Table 2**

Aging heat-treatment applied to the single-crystal superalloy (A.C. air cooled) and resulting average  $\gamma'$  sizes and their volume fractions.

No. of samples	Aging heat-treatment	Average $\gamma'$ size (nm)	Volume fraction (%)
N-1	1000 °C/4 h/A.C.+870 °C/25 h/A.C.	340	68.4
N-2	1100 °C/4 h/A.C.+870 °C/25 h/A.C.	420	69.5
N-3	1180 °C/0.5 h/A.C.+1150 °C/4 h A.C.+870 °C/25 h/A.C.	513	70.0



**Fig. 1.** The morphologies of  $\gamma'$  precipitates of superalloys after various aging rules: (a) N-1 sample, (b) N-2 sample and (c) N-3 sample.

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