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# High temperature creep properties of directionally solidified CM-247LC Ni-based superalloy



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#### ARTICLE INFO

# Article history: Received 5 August 2015 Received in revised form 26 December 2015 Accepted 30 December 2015 Available online 31 December 2015

Keywords: Ni-based superalloys High-temperature creep Cooling rates TEM

#### ABSTRACT

This study explores the effects of cooling rate after solution heat treatment on the high temperature/low stress (982 °C/200 MPa) creep properties of CM-247LC Nickel base superalloy. Cooling rate was controlled by blowing argon gas, air cooling, and furnace cooling, which, in turn, gave rise to corresponding cooling rates (from 1260 °C to 800 °C) of 18.7, 7.4, and 0.19 °C/s, respectively. The results indicated that higher cooling rate from the solution heat treatment temperature led to finer  $\gamma'$  precipitates and much improved tertiary creep as well as rupture life time in high-temperature creep test. The microstructural analyses using both scanning electron microscopy (SEM) and transmission electron microscopy (TEM) revealed that finer  $\gamma'$  precipitates and narrower  $\gamma$  channel width could result in denser rafting structure which might have hindered the climb of dislocations across the precipitates rafts.

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#### 1. Introduction

Owing to their excellent high temperature strength and oxidation resistance, which are required for the hot section of highefficiency turbine engines operating at stringent temperature (usually at  $T \ge 0.7$   $T_m$ , with  $T_m$  being the melting temperature of the alloy) and oxidizing environment, Ni-based superalloys have received extensive attention, recently [1-14]. Among them, directionally solidified CM-247LC Ni-based superalloy has prompted tremendous research interest because of its superb high temperature properties [2,4,5,15,16]. Since the superalloys are primarily strengthened by the ordered coherent Ni<sub>3</sub>Al-type  $\gamma'$  precipitates existing in the  $\gamma$  matrix, controlling the volume fraction as well as the size and morphology distribution of the  $\gamma'$  precipitates is thus of essential importance to improve the hightemperature properties [2,3,6-8,10-12,14-19]. As a result, in addition to tuning to the concentration of the prominent solutes, such as Al and/or Ti, carefully manipulating the manufacturing and subsequent heat treatment processes has been playing an important key in optimizing the high temperature mechanical properties of these alloys [2,7,11,14-16,18-19].

Although in industrial production practices blowing argon gas, air cooling, and furnace cooling were commonly used as part of

the heat treatment protocol, however, it is noted that there have been only very limited published literature specifically devoted to address the possible impacts resulting from the cooling rate on the eventual high-temperature mechanical properties of the Ni-based superalloys [4,5,16]. Some studies had briefly mentioned the importance of the cooling rate after solution heat treatment, but no details have been described [20–21].

In this study, we conducted systematical investigations to delineate the effects of post solution heat treatment cooling rate on the high-temperature mechanical properties of directionally solidified CM-247LC Ni-based superalloy. In particular, the correlations between the cooling rate-induced microstructure changes and the high-temperature creep behaviors are addressed.

#### 2. Experimental details

The CM-247LC Ni-based superalloy ingots used in this study were purchased from Cannon-Muskegon Co. (MI, USA). Directionally solidified (DS) CM-247LC round bar specimens with 12 mm in diameter were cast from in-house Bridgeman type [001] vertical tube furnace at a withdrawal rate of 180 mm/h under  $10^{-1}$  Torr vacuum.

Solution heat treatment was conducted at 1260 °C for 2 h, then cooling down to room temperature with three different cooling rates, namely, blowing argon gas cooling, air cooling and furnace cooling. The corresponding cooling rates (from 1260 °C to 800 °C)

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were estimated to be 18.7, 7.4, and 0.19 °C/s for argon, air, and furnace cooling, respectively. Subsequently, all the samples were subjected to the same aging process with the following detailed heat treatment scheme. Firstly, the samples were aged at 1079 °C for 4 h followed by argon cooling to room temperature. The second aging process was carried out at 871 °C for 20 h followed again by argon cooling to room temperature. Creep tests were performed at 982 °C with a constant stress of 200 MPa (982 °C/200 MPa) using an ATS Series 2330 lever arm creep tester produced by Applied Test Systems Inc. According to the Japanese Industrial Standard (JIS Z 2271) [22], the dimensions of rod specimens were machined to a diameter of 6.35 mm with a gage length of 25.4 mm. A Hitachi field-emission scanning electron microscope (SEM) equipped with a Horiba energy-dispersive X-ray spectrometry (EDS) system was used to examine the microstructure of samples after each heat treatment process and the cross section after creep test. FEI Tecnai G<sup>2</sup> 20 S-Twin and FEI E.O Tecnai F20 G<sup>2</sup> MAT S-TWIN field emission gun transmission electron microscope (TEM) were used to observe the dislocation morphology of each sample after creep test.

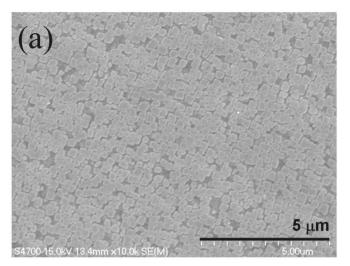
In the present work, the volume fraction of  $\gamma'$  was determined from multiple sets of 10,000X SEM photos by using image analysis software, MA-Pro following the ASTM E562-08 standard [23]. The width of  $\gamma$  channel was measured by TEM images because of their high magnifications allowed us to determine the width more precisely.

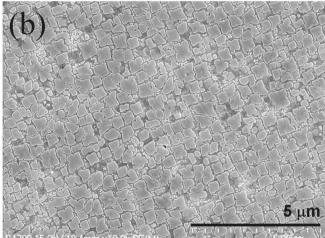
#### 3. Results and discussion

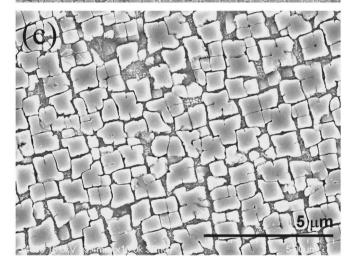
#### 3.1. Microstructures after heat treatments

Fig. 1 shows the SEM photographs taken for samples right after solution heat treatment with different cooling rates to reveal the effect of cooling rate on the microstructure of samples. It is evident from Fig. 1 that, although the appearance of carbides and  $\gamma - \gamma'$ eutectic phase distribution looks very similar, depending on cooling rate, the detailed microstructure has already displayed distinct differences even at this stage. For instance, in addition to the size of  $\gamma'$  precipitates, the morphology of the  $\gamma'$  precipitates appears to remain relatively intact for the rapidest cooled sample (Fig. 1(a)). In contrast, with slower cooling rates after solution heat treatment, the  $\gamma'$  precipitates grew larger (shown in Fig. 1(b) and (c)). Previously, it has been conceived that splitting of the  $\gamma'$  precipitates could refine the microstructure are beneficial for the stability of the alloy under thermo-mechanical load. However, as pointed out very recently by Vogel et al. [24] that the splitting of the  $\gamma'$  precipitates driven by nucleation of  $\gamma$  inside  $\gamma'$  precipitates due to the super-saturation of Ni is, in fact, detrimental to the thermo-mechanical properties. Therefore, in order to improve the stability of the microstructure and hence the thermo-mechanical properties of the alloy, it is desirable that the  $\gamma'$  precipitates should not be split by the  $\gamma$  particles (or plates), but instead remain intact during heat treatment. Indeed, as will be seen below, the samples obtained by argon gas cooling from the solution heat treatment exhibit much better thermo-mechanical properties than those obtained through slower cooling rates. It is suggestive that the ascooled microstructure immediately after solution heat treatment might have already played a decisive role in determining the eventual thermo-mechanical properties of these alloys.

Fig. 2 reveals the evolved microstructure of the respective samples after the full two-step aging processes. As can be seen from the SEM photograph shown in Fig. 2(a), for the argon cooling specimens, the  $\gamma/\gamma'$  structure becomes more distinctive, *i.e.* the morphology of the  $\gamma'$  precipitates and  $\gamma$ -channels are clearly distinguishable as compared to that seen in Fig. 1(a). Furthermore,







**Fig. 1.** SEM photographs showing the as-cooled microstructure of CM-247LC DS specimens after solution heat treatment: (a) argon cooling, (b) air cooling, and (c) furnace cooling.

some coalescence of  $\gamma'$  precipitates is also observable. Fig. 2 (d) shows the corresponding TEM photograph of the same argon cooled sample; revealing the coherent nature of the  $\gamma/\gamma'$  structure (please see the inset diffraction pattern in Fig. 2(d)). The size of  $\gamma'$  precipitates of argon cooling specimens is about 0.3–0.5  $\mu$ m. For the air-cooled specimens (Fig. 2(b) and (e)), it is apparent that, although the size of the  $\gamma'$  precipitates (about 0.3–0.7  $\mu$ m) is not

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