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Materials Characterization



Effect of minor carbon additions on the high-temperature creep behavior of a single-crystal nickel-based superalloy



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ABSTRACT

Different amounts of carbon were added to a single-crystal nickel-based superalloy. The microstructural evolution of these alloys before and after high-temperature creep tests was investigated by employing scanning electron microscopy and transmission electron microscopy. Upon increasing the carbon contents, the volume fraction and diameter of the carbides increased gradually: however, the creep lives of the alloys increased slightly at first and subsequently decreased. The formation of second-phase particles, such as the nano-sized $M_{23}C_6$, blocky and needle-shaped μ phase, was observed in the creep samples, which was closely related to the high-temperature creep behaviors.

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1. Introduction

Single-crystal (SX) superalloys are widely used in aircraft engines and land-based gas turbines because of their excellent hightemperature strength and oxidation resistance. Further improvements in the temperature capabilities of SX superalloys have been achieved with the addition of higher levels of refractory elements, such as tantalum, rhenium and tungsten [1]. However, the casting characteristics of these modern high-refractory alloys are significantly worse than those of earlier alloys [2,3]. Carbon has recently been reintroduced to these alloys to improve the castability [4–6], and therefore, more attention has been focused on the effect of the carbon additions on microstructures and mechanical properties of SX superalloys [7–16].

In general, the addition of carbon may increase the volume fraction of carbides [8–10,14,15], minimize the casting pores [8,10,11], reduce the casting/solidification defects [5,7,13], and enhance the tolerance angle of low-angle grain boundaries [8]. Few works in the literature focus on the effect of the carbon addition on mechanical properties [8,11,12]. It was reported that a carbon addition (0.05 wt.%, 0.1 wt.%) will reduce the principal crack initiation sites, namely casting pores, because of the precipitation of the MC carbides during solidification. This phenomenon improves the rupture life at 850 °C, whereas the high-temperature performance of the modified alloys at 1050 °C was reduced by the presence of retained eutectic regions due to incomplete solidification [8]. Liu et al. reported that longer creep rupture lives were obtained at 1038 °C/172 MPa with the addition of 0.015 wt.% and 0.05 wt.% carbon, which was interpreted to be due to the combined effects of the beneficial effect of the $(M_6C)_2$ carbides and the reduction of casting pores with increasing carbon content [11,17]. Additionally, in most of the work, over 0.05 wt.% carbon was added, which causes the formation of script carbides and leads to the consumption of refractory elements; these effects influence the microstructure and the creep behavior. Therefore, it is necessary to clarify the effect of minor carbon additions on the creep properties of SX superalloys.

The purpose of this study was to evaluate the effect of minor carbon additions on the high-temperature creep properties of a SX superalloy. Unlike the previous study, minor carbon additions (less than 0.03 wt.%) were added to a second-generation SX superalloy in the present experiment. The creep rupture lives were compared, and the microstructural evolution after creep was investigated.

2. Experimental procedure

The nominal composition of the experimental alloy is 5Cr, 10Co, 6W, 2Mo, 6Al, 3Re, 9Ta, 0.1Hf and the remainder Ni (wt.%). A master alloy free of carbon was initially cast using a vacuum induction melting furnace, and then, different amounts of carbon were added to the master alloy during directional solidification using the liquid metal cooling (LMC) method. The SX bars with longitudinal orientation within 10° from [001] were used in the present experiment. The resulting alloys

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with actual carbon contents of 0.005 wt.%, 0.013 wt.% and 0.032 wt.% were named A, B and C, respectively.

SX bars with different carbon contents were heat treated (solution heat treated at 1310 °C for 5 h, followed by two aging steps at 1150 °C for 4 h and 870 °C for 24 h). Some heat-treated SX bars were cut vertically to the DS direction. Five BSE micrographs with areas of $300 \times 400 \,\mu\text{m}$ were acquired from each sample at a magnification of $500 \times$ to minimize the standard deviation, and the size and volume fraction of the carbides in each alloy were analyzed.

The partitioning behavior of the main elements was determined using an electron probe micro-analyzer (EPMA) on fine polished but unetched samples. Point scans in the dendrite cores and interdendritic regions (next to the γ/γ' eutectics but not the areas of carbides) were performed. A 5-µm spot size was used for all the EPMA characterization to simultaneously sample both the γ and γ' phases and obtain a more representative average composition. The partitioning coefficient, k', of each element was identified by dividing the weight percent of each element at the dendrite cores by that in the interdendritic regions. Fifteen points from each area were averaged to reduce variation.

Creep specimens were machined from the heat-treated SX bars along the [001] direction with a gauge length of 25 mm and a gauge diameter of 5 mm. Creep tests were conducted at 1100 °C and 152 MPa. Three samples for each alloy condition were tested, and the results reported are the average of the three tests. One group of the creep ruptured samples was cut along the diameter of the gauge section parallel to the load axis for SEM observation; transverse sections approximately 3 mm from the fracture surface were also examined. The other group of samples was prepared for transmission electron microscope (TEM) investigation. Thin discs were cut normal to the load axis and approximately 5 mm apart from the fracture surface. These discs were mechanically grounded to 45 μ m and electro-polished at -20 °C in a solution of 10% perchloric acid and 90% ethanol. Selected area diffraction (SAD) patterns were indexed for the phase determination.

3. Results and discussion

3.1. As-cast and heat-treated microstructure

3.1.1. General microstructure of the as-cast and heat-treated samples

The morphologies of the as-cast and heat-treated samples are shown in Fig. 1. Large γ/γ' eutectics in the interdendritic regions (Fig. 1a) were observed in the as-cast samples; there are small cubic γ' in dendrite cores (Fig. 1b, left) and large irregular γ' in the interdendritic regions (Fig. 1b, right). After full heat treatment, the γ/γ' eutectics dissolved completely (Fig. 1c), and the microstructure of these alloys is composed of a matrix containing well-aligned coherent cuboidal γ' precipitates, with an average edge length of approximately 0.4 µm (Fig. 1d).

Fig. 2 shows the carbide morphology in the heat-treated alloys. Almost no carbides could be observed in alloy A (Fig. 2a), only a small amount of carbides could be detected occasionally in alloy B (Fig. 2b), and a large volume fraction of blocky carbides was observed in alloy C (Fig. 2c). These carbides were confirmed to be MC carbides containing Ta. The decomposition of carbides was not detected in the heattreated alloys.

The average diameter and volume fraction of the carbides in the experimental alloys were metallurgically analyzed, and the results are presented in Table 1. Only 0.24% carbides exist in alloy C, and the carbide diameter doubled from 0.63 μ m to 1.24 μ m when the carbon content increased from 0.013 wt.% to 0.032 wt.%.

3.1.2. Micro-segregation in the heat-treated samples

After solution heat treatment, the partitioning ratio of elements in different alloys was measured (Fig. 3). The results indicate that with the carbon content increasing from 0.005 wt.% to 0.032 wt.%, the partitioning ratios of Re and Mo decreased slightly. More Cr was detected in the interdendritic regions than in the dendrite cores with increasing carbon content. Whereas Ta concentrated in the interdendritic regions of alloy A, in alloy C, Ta was enriched in the dendrite core



Fig. 1. OM and SEM micrographs of the as-cast and heat treated samples. (a) OM image of the as-cast sample, (b) fine γ' in dendrite core (on the left) and irregular γ' in interdendritic regions (on the right), (c) OM image and (d) SEM image of the full heat-treated samples.

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