Effect of Long-Term Thermal Exposures on Microstructures and Mechanical Properties of Directionally Solidified CM247LC Alloy

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(received date: 5 September 2012 / accepted date: 17 January 2013)

A directionally solidified CM247LC alloy was exposed at 871 °C and 982 °C for 1000 h, 5000 h, and 10000 h under free stress in order to study the effect of microstructural degradation on the creep properties. None of the specimens exposed at temperatures up to 10000 h produced any kind of topologically close-packed phases because of the excellent phase stability of CM247LC alloy. The plate-like M6C carbide was formed only at exposure of 982 °C for 10000 h through a decomposition reaction between γ and MC. Moreover, an M23C6 carbide layer was observed between the M6C and the matrix. The exposure at 982 °C for 5000 h and 10000 h had a spontaneous rafting of γ′ under free stress, while the exposure at 871 °C for 1000 h, 5000 h, and 10000 h had a non-rafted structure. The spontaneous rafted structure resulted in a drastic decrease in creep life. A 3-dimensional morphology of γ′ in the as-crept specimens, which were pre-exposed at 982 °C for 5000 h and 10000 h, had a non-rafted structure. This microstructural feature proves that the significant decrease in creep life of the specimen resulted from a loss of coherency between γ and γ′.

Key words: alloys, aging, microstructure, tensile test, Ni-base superalloys

1. INTRODUCTION

Various kinds of directionally solidified (DS) Ni-base superalloys have been designed and evaluated for the main application as dynamic parts of high efficiency industrial gas turbines (IGTs), which require an excellent combination of mechanical properties and environmental resistance at high temperature [1-5]. Fulfilling the requirements for the application of a DS Ni-base superalloy produced alloys rich in Cr and containing B, C, Zr, and Hf as minor elements; Cr is added to improve corrosion resistance, and B, C, Zr, and Hf are added to strengthen grain boundaries [6]. The concentrations of Cr and the minor elements must be controlled carefully because additions of Cr, B, Zr, and Hf give rise to adverse effects in liquidus temperature and phase stability and eventually a decrease in creep properties. The start-up, rotation of turbine and shut-down from high temperature to room temperature exposes a designed alloy to a severe environment, such as high temperature, varying stress conditions, and corrosive atmosphere. A careful understanding of the degradation of the microstructure and mechanical properties of a DS superalloy under their combined surroundings is required for managing the life of dynamic parts made of a DS superalloy in service.

In modifying a DS Ni-base superalloy and in managing the life of components in service it is important to understand the microstructural degradation at high temperature during the service. The standard heat treated DS superalloy undergoes three types of microstructural degradation during isothermal exposures. First, the L12 structure of coherent precipitates γ′ undergoes coarsening, a morphological variation from the initial cuboidal structure into a spherical one or rafted one [6]. Second, the topologically close-packed phases (σ, μ, p, R, etc.) nucleate and grow consuming the refractory elements (W, Mo, Ta, etc.) of γ and γ′ [6]. Third, MC carbides react with the matrix and decompose into M23C6 and M6C, which results in the morphological variation of the carbides and composition of the matrix [4,6]. The microstructural and compositional variation induced by high temperature exposure essentially deteriorates the high temperature mechanical properties of a DS superalloy. Therefore an entire understanding of the growth kinetics of γ′ and the TCP phases and carbides at various temperatures is essential for managing the life of and improving a DS superalloy.

The degradation behaviors of various DS superalloys have
been studied in a distinct manner [4,7-12]. Choi et al. [4] investigated the microstructural evolution of equiaxed GTD111 during isothermal exposure and reported that the growth rates of $\gamma'$ complying with Ostwald ripening were $1.030 \times 10^{-5}$ $\mu m^3/h$, $4.134 \times 10^{-5}$ $\mu m^3/h$, and $10.060 \times 10^{-5}$ $\mu m^3/h$ at 871 °C, 927 °C, and 982 °C, respectively. They also reported that the MC carbide decomposed into $\eta$ and $M_2C_6$ at 871 °C [4]. The effect of pre-exposure with and without loading at high temperature on the fatigue life of a DS GTD 111 was studied by Gordon et al. [9], and they suggested that the most detrimental pre-exposure configuration was a sustained tensile load at high temperature. Choi et al. investigated the isothermal degradation behavior of an equiaxed CM247LC and reported that W-rich needle-like $M_6C$ and blocky $M_2C_6$ formed through the decomposition of scriptal and blocky MC, respectively [12].

It is known from previous studies as mentioned above that the degradation behavior is sensitive to the exposure conditions, temperature and loading, and the composition of an alloy. However the microstructures exposed at various temperatures and times and the related creep properties of a DS CM247LC have not been studied to date.

In this study the columnar grained DS CM247LC alloy was exposed at isothermal temperatures of 871 °C and 982 °C for durations from 1000 h to 10000 h in order to study the effect of the as-exposed microstructure on the creep properties. The main microstructures that we investigated were the formation kinetics of TCP, the decomposition behavior of MC resulting from a reaction with the matrix, and the morphological variation and growth behavior of $\gamma'$ as a function of exposure temperature and time.

2. EXPERIMENTAL PROCEDURES

The ingot of CM247LC alloy (0.07 C, 8.1 Cr, 9.2 Co, 0.5 Mo, 9.5 W, 3.2 Ta, 5.6 Al, 0.7 Ti, 0.015 B, 0.015 Zr, 1.4 Hf, and the balance of Ni in wt%) was supplied by Cannon-Muskegon corporation. The master ingot was directionally solidified with a withdrawal rate of 4 mm/min into a round bar 13 mm in diameter and 270 mm in height. The grain shape was controlled to be columnar using a directional solidification furnace of ALD ISP 0.5 DS/SC/LMC. The directionally solidified bar was subjected to the standard heat treatment as recommended by the supplier; solutionized by 4-step heating up of 1221 °C for 2 h + 1232 °C for 2 h + 1234 °C for 2 h with a ramping rate of 0.5 °C/min + 1250 °C for 2 h with a ramping rate of 0.5 °C/min + water quenching (WQ) followed by 2-step aging at 1080 °C for 4 h + air cooling (AC) and 871 °C for 20 h + AC. The standard heat treated specimens were exposed at 871 °C and 982 °C for 1000 h, 5000 h, and 10000 h. The creep properties of the specimens subjected to standard heat treatment and thermal exposures were evaluated by an ATS creep tester. The stresses and temperatures of the creep tests were 760 °C, 770 MPa and 927 °C, 287 MPa and 982 °C, 205 MPa.

The samples for optical microscopy were polished and etched with Kalling's reagent (2 g CuCl$_2$ + 40 ml HCl +80 ml ethanol) to dissolve the $\gamma'$ phase. Electrolysis etching which dissolves the $\gamma$ phase was performed with a solution of 3 g CrO$_3$ + 100 ml H$_2$O to observe the morphology of $\gamma'$ before and after the creep test using a scanning electron microscope, a JEOL Model 5800, at an accelerating voltage of 20 kV. The feret diameters of $\gamma'$ with exposure temperature and time were measured using an image analyzer.

The specimens for a transmission electron micrograph were grounded into 70 µm and subjected to electrochemical polishing with a solution of methanol + 20% perchloric acid under -20°C at the voltage of 22 V. A JEM-2100F model transmission electron microscope was used at an accelerating voltage of 200 kV.

3. RESULTS AND DISCUSSION

3.1. Microstructural evolution as a function of thermal exposure

The extended isothermal exposure at high temperature of a DS nickel-base superalloy is well known to result in the morphological variation of $\gamma$ and $\gamma'$, and the formation of topologically close-packed phases (TCP), and compositional and structural changes of carbides. The high temperature mechanical properties depend upon not only the morphology and volume fraction of coherent precipitates $\gamma'$ but also the content of low diffusivity refractory elements in $\gamma$ and $\gamma'$. The formation of TCP phases and compositional and structural changes of carbides results in depletion of refractory elements in $\gamma$ and $\gamma'$. Therefore, the morphology of $\gamma'$ and the formation kinetics of TCP and carbides during the service is a major interest in the design and evaluation of a superalloy.

3.1.1. Formation of topologically closed-packed phases and carbides

Figure 1 shows the optical and secondary electron micrographs of the standard heat treated DS CM247LC alloy. Eutectic structures, carbides, and well-aligned eutectic carbides were observed. The standard heat treated DS CM247LC alloy was exposed at the temperatures of 871 °C and 982 °C for 1000 h, 5000 h, and 10000 h. The specimen exposed up to 10000 h did not bear any kind of TCP phases such as $\sigma$, $\mu$, $\eta$, R, or Laves phases at those temperatures as shown in Fig. 2. Yukawa et al. [13] suggested that the Md value (average d-orbital level of alloying element) might be used as an alloy parameter to estimate the phase stability. They showed that the limit of the Md value for TCP might be 0.987 for the alloys containing 10-13 at% Cr. In addition, a lower content of Cr might increase the limit of Md. The Md value of CM247LC alloys containing 9.3 at% Cr was