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On the precipitation hardening of the directionally solidified GTD-111 Ni-base superalloy: Microstructures and mechanical properties

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ABSTRACT

Effects of precipitation hardening on microstructures and mechanical properties of the directionally-solidified (DS) Ni-base GTD-111 superalloy are investigated. DS specimens were subjected to different cycles of solution treatments at temperatures of 910 °C, 980 °C and 1050 °C. The solidified and heat-treated microstructures along with the stress rupture properties were evaluated to investigate influence of the homogenization and second solution treatments. The results showed the homogenization treatment resulted in a more uniform distribution of the γ′ precipitates. It was shown that growth and preferred distribution of the γ′ precipitates occurred along the < 100 > direction in the primary solution treatment. More growth of γ′ precipitates during secondary solution treatment led to the splitting of the precipitates into a group of fine precipitates. The best stress rupture result was achieved in the specimen with the second solution treatment at 980 °C for 2 h followed by aging at 843 °C for 24 h due to the suitable size, volume fraction, and size distribution of the γ′ precipitates in the DS microstructure.

1. Introduction

Ni-base superalloys are frequently used for the manufacture of turbine blades of aero-engines and land-based gas turbines, where high-temperature mechanical properties and corrosion resistance are needed [1–3].

Strengthening of Ni-base superalloys which is required to establish the acceptable high-temperature properties, is usually considered by two main strengthening mechanisms of solid solution strengthening and precipitation hardening [4]. These strengthening mechanisms in γ′ strengthened superalloys depend on alloying elements distribution in matrix, morphology and contents of the γ′ phase and the γ-γ′ eutectic resulted from the last stages of the alloy solidification [2,5]. Precipitation hardening is usually conducted in two essential stages of solution and aging treatments (ATs) through which dissolution of γ′ precipitates for further precipitating, and removal or reduction of solidification segregation are aimed to occur [6].

DS superalloys are usually used as the blading material in advanced gas turbine engines [7,8]. Directional solidification techniques enable the solidification structure of materials to align in < 001 > texture parallel to the axis of the blade [9,10]. This structure improves thermal fatigue resistance because the < 001 > direction with minimum Young modulus results in reduced thermal stresses [4,10]. In addition, as grain boundaries are failure initiation sites, the alignment or elimination of the grain boundaries normal to the stress axis would increase creep strength and ductility at elevated temperatures [4,8].

GTD-111 is a Ni-base superalloy commercially used in manufacturing of the first stage gas turbine blades in the power plant gas turbines [1,11,12]. The GTD-111 has a chemical composition and microstructure near to the Rene 80 and IN738LC [1,12,13]. This microstructure is composed of dendritic γ matrix with γ′ precipitates with a bimodal size distribution, γ–γ′ eutectic, MC and $M_{23}C_6$ carbides, and a small amount of detrimental phases like σ, δ, η and Laves [13,14]. Chemical analysis indicated that MC carbides are rich in Ti, W and Ta, while $M_{23}C_6$ type carbides contain Cr and Mo [1,2,14]. Yang et al. [1] have reported that both MC and $M_{23}C_6$ carbides promote stress rupture properties of this alloy by decreasing dislocation movement and grain boundary sliding.

Some investigation showed that homogeneity of the microstructure and uniform distribution of γ′ particles would lead to the improvement of high temperature properties of DS Ni-based superalloys [15,16]. The homogenization treatment (HT) which is normally conducted at or above 1200 °C, reduces segregation of alloying element caused by solidification [2]. Trexler [17] reported that the best temperature of γ′ solution temperature is 1200 °C which has been obtained by three methods of dilatometry, heat treatment/metallography and differential thermal analysis (DTA). Superior to this temperature may cause local melting and unwanted effects on microstructure and mechanical

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properties [2]. The subsequent primary solution treatment (PST) improves high temperature tensile strength of DS Ni-based superalloy by increasing rafting of γ′ precipitates so that dislocations can no longer climb around the lamellar γ′ structure [15]. It is recently reported that the application of a second solution treatment (SST) may also improve the stress rupture life of GTD-111 Ni-base superalloy through increasing in γ′ phase nucleation and volume fraction [1]. In the present study, effects of the HT and SST stages at different temperatures on microstructures and high-temperature mechanical properties of the DS Ni-base superalloy GTD-111 are investigated. The improvement of the rupture life of the heat-treated alloys is discussed based on microstructural features.

2. Materials and experimental procedures

Several cylindrical DS rods with the dimension of 15 mm in diameter and 110 mm in length were solidified using a home-made Bridgman furnace. The melt temperature was 1500 °C and the withdrawing rate was 6 mm/min. The chemical composition of the investigated Ni-based superalloy GTD-111 was determined by X-ray fluorescence (XRF) and optical emission spectroscopy (OES), as presented in Table 1.

The critical temperatures including the liquidus and solidus, the solvus of the precipitate phase γ′ and γ-γ′ eutectic were determined by differential scanning calorimetry (DSC) technique (NETZSCH STA 409 PC/PG model). Test pieces were machined from the DS rods with 3 mm diameter, 5 mm height and 5 mg weight. The DSC test was conducted at 1400 °C under helium inert gas (purity of 99.999%) with the heating rate of 5 K/min. The results of the DSC test are shown in Table 2. According to Table 2, the homogenization temperature was considered at 1200 °C.

The DS specimens were cut along the longitudinal and transverse sections with respect to the solidification direction and subjected to five different heat treatment cycles as shown in Table 3. In all these heat treatment cycles, the specimens were heated to the specified temperatures with the rate of about 10 K/min in argon atmosphere followed by air cooling to room temperature.

All cast and heat treated specimens were mounted and grounded using silicon carbide papers and polished till 4000 grit finish followed by polishing with 0.3 and 0.05 μm alumina suspension. The specimens were then etched using the macro-etch solution of 25 g $FeCl_3$+27 mL HCl+40 mL H_2O at 60 °C for 20 min and the micro-etch solution of 33-mL H_2O+33-mL CH_3COOH+33-mL HNO_3+1-mL HF at room temperature for 8 s. Microstructures were characterized using optical microscopy (OM, Nikon EPIPHOT 300) and scanning electron microscopy (SEM, Philips XL30) techniques. The size and volume fraction of phases were obtained by Image J software (V 1.48, US National Institute of Health). The volume fraction, average size and size distribution of the γ′ precipitates were calculated in the surface area of interest.

Standard stress rupture specimens were machined from the heat treated DS bars in the longitudinal direction. The quality of the specimens was checked by a radiographic test to ensure the absence of cracks and voids. Stress rupture tests were carried out at 186 MPa and 982 °C using E101RQ13 stress rupture testing machine on round bar specimens with a gauge length of 20 mm and diameter of 4 mm according to ASTM E8M [18] and ASTM E139M [19] standards.

Table 2
Transition temperatures for GTD-111 (°C).

γ′ solvus	solidus	γ-γ′ eutectic	liquidus
1175	1295	1325	1340

Table 3
Heat treatment cycles of the DS specimens.

Specimen code	Homogenization Treatment (HT)	Primary Solution Treatment (PST)	Secondary Solution Treatment (SST)	Aging Treatment (AT)
C1	–	1120 °C/2 h	–	843 °C/24 h
C2	1200 °C/2 h	1120 °C/2 h	–	843 °C/24 h
C3	1200 °C/2 h	1120 °C/2 h	1050 °C/2 h	843 °C/24 h
C4	1200 °C/2 h	1120 °C/2 h	980 °C/2 h	843 °C/24 h
C5	1200 °C/2 h	1120 °C/2 h	910 °C/2 h	843 °C/24 h

Fig. 1. The macrograph of the as-cast longitudinal grain structure of the DS specimen. The solidification direction is left to right.

3. Results and discussion

3.1. Microstructural analysis

3.1.1. Cast microstructures

The macrograph of the DS GTD-111 specimen is shown in Fig. 1. During directional solidification, a columnar-grained structure is provided by temperature gradient which is achieved by the water cooled chill plate at the bottom of the mold. As shown in Fig. 1, near the water cooled chill plate, the structure consists of the equiaxed grains as a result of the high temperature gradient. With increasing distance from the chill plate and decreasing the thermal gradient, directional and competitive growth of dendrite occurred in [001] direction, resulting in a decrease in grain density.

The dendritic microstructures of the DS specimen in both longitudinal and transverse cross sections are illustrated in Fig. 2. The average values of the primary dendrite arm spacing (PDAS) in the transverse section and the secondary dendrite arm spacing (SDAS) in the longitudinal cross section were calculated as 207 ± 7 μm and 53 ± 2 μm, respectively. During the dendritic competitive growth, the PDAS is gradually increased as a result of the reduction in thermal gradient. Although the reduction of thermal gradient during solidification could result in a longer solidification time, and the SDAS is related to the local solidification time, the present data show that the SDAS keeps approximately unchanged along the DS rod. The PDAS and SDAS values of the DS GTD111 superalloy were changed through the specimen according to the Eqs. (1) and (2), respectively:

$$PDAS = 0.1796x + 196.79 \quad (1)$$

$$SDAS = -0.0596x + 56.123 \quad (2)$$

where x is the distance from the chill plate in mm and PDAS and SDAS are dendritic arm spacing in micron.

The microstructure of the DS specimens includes γ′ precipitate, MC

Table 1
Chemical compositions of the GTD-111 superalloy (wt%).

Element	Cr	Co	Al	Ti	W	Mo	Ta	Cu	S	P	Ni
wt%	13.5	9.41	3.4	4.88	3.69	1.68	3.9	0.012	0.001	0.003	Bal.