Effect of boron on the grain boundary morphology and creep property in nickel-based superalloy LESS 1

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To examine the correlation between microstructure and creep property by addition of boron, effect of boron on the grain boundary morphology and creep resistance in nickel-based superalloy LESS 1 is investigated. Scanning electron microscopy (SEM), transmission electron microscopy (TEM) and small-punch creep test are carried out. In case of boron-added LESS 1, so-called boro-carbides (M₂₃(C, B)₆) and γ ` film (Ni₃(Al, Ti)) are precipitated at grain boundary (GB). Also creep resistance is considerably increased by addition of boron. The improvement of creep resistance can be attributed to the change of grain boundary morphology by addition of boron.

Keywords: Nickel-based superalloy, creep, boron, grain boundary, boro-carbide

1. Introduction

Nickel-based superalloys are under development as next generational materials for Hyper-Super Critical fossil power plant because of its outstanding mechanical properties at high temperature [1] and it has been proved that the additions of boron improves creep resistance of nickel-based superalloy by previous study. However, the mechanism about reinforcement have not been systemically studied by other research groups. Among them, the main explanations are divided. First, boron atoms are distributed at γ -M₂₃C₆ interface and this interface disrupts the movement of dislocation [2, 3]. Second, boron atoms are soluble in lattice of Ni₃Al and produce the strengthening effect [4, 5]. Finally, the reaction of carbide precipitation is influenced by boron [6]. As mentioned above, understanding rules of boron in nickel-based superalloy is not confirmed.

In this research, to understanding the role of boron, the existent position of boron and phase transition in nickel-based superalloy LESS 1 were investigated.

LESS 1 alloy which designed by changing the composition of Cr, Al, Ti and W for control of TCP phases of INCONEL alloy 740 was used in this study. Its nominal chemical composition is Ni-22Cr-20Co-2Nb-2W-1.5Al-1.5Ti-0.3C (wt. %). The alloy was prepared by vacuum induction furnace. The experimental alloys LESS 1 and LESS 1 (B) are without and with B addition respectively. These specimens were homogenized at 1200 °C for 16 h and hot-forged at 1100 °C. The as-forged specimens were subjected to standard heat treatment: 1200 °C/0.5 h WQ + 800 °C/16 h AQ. Before starting investigation, actual chemical composition of LESS 1 and LESS 1 (B) was confirmed by EDS, OES and carbon/sulfur analyzer. Boron contents were 10 ppm and 110 ppm respectively.

Microstructure was observed by 7600F (JEOL) Field Emission Gun Scanning Electron Microscope (SEM) and JEM ARM 200F (JEOL) Transmission Electron Microscope (TEM).

Creep tests were carried out at 700 °C/50 kgf by a small-punch creep tester. The sample size was $10 \times 10 \times 0.5$ mm.

3. Results and discussion

3.1. Microstructure characteristic

Grain size and grain interior between LESS 1 and LESS 1 (B) were similar. Fig. 1 shows the typical GB morphology of LESS 1 and LESS 1 (B). In case of LESS 1, Ti and Nb-enriched MC and Cr-enriched $M_{23}C_6$ carbides were partially distributed at the GB, whereas LESS 1 (B) revealed that Cr-enriched $M_{23}C_6$ carbides were continuously distributed at the GB. In general, the critical location of $M_{23}C_6$ carbides can improve the rupture strength. But rupture failure is usually initiated by desorption of γ - $M_{23}C_6$ interface [7].



Fig. 1 SEM images showing the grain boundary morphology.

Results of TEM investigation in LESS 1 (B) are shown in Fig. 2. By the EDS analysis, coexisting titanium and aluminum which surrounding Cr-enriched carbides was observed (Fig. 2a). To identify the phase, SADP analysis was carried out and indicated that γ ` film formed along the GB (Fig. 2b) which is believed to optimize the rupture properties [7].



Fig. 2 TEM results of LESS 1 (B): (a) EDS, (b) dark field image of γ ` film.

Fig. 3 shows distributions of carbon and boron by EELS. It confirmed that the $M_{23}C_6$ -type carbides in the γ ` film were not $Cr_{23}C_6$ but $Cr_{23}(C, B)_6$.



Fig. 3 Result of EELS showing the distribution of boron and carbon at the GB.

3.2 Creep behavior of LESS 1 and LESS 1 (B)

The result of creep tests is illustrated in Fig. 4. It is to be noted that in case of LESS 1 (B), test was aborted. Although test of LESS 1 (B) was aborted, it was confirmed that creep resistance of LESS 1 (B) was significantly improved.



Fig. 4 Result of small-punch creep test at 700 °C/50 kgf.

4. Conclusion

Based on the results presented in this study, the following conclusions can be made:

- After standard heat treatment, grain size and grain interior of LESS 1 and LESS 1 (B) are similar but reaction of carbide precipitation is changed by boron.
- Source for Cr-enriched M₂₃C₆ is not only carbon but also boron, so that phase fraction of M₂₃C₆ is increased by addition of boron and it should be noted that the M₂₃C₆ mainly precipitates at grain boundary.
- 3) As a result of increased $M_{23}C_6$ carbides, concentrations of aluminum and titanium in γ matrix are simultaneously increased, so that it is supposed that γ ` film is formed surrounding $M_{23}C_6$ carbides.
- Creep resistance is considerably increased by addition of boron and it is assumed that the changes of grain boundary morphology make creep resistance improvable.

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