Improvement of Creep Resistance in Wrought Nickel Based Superalloys by Addition of Trace Elements

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The alloys used for fossil fuel power plants are transitioning from stainless steels that operate below 600 $^{\circ}$ C to nickel based superalloys that can operate up to 700 $^{\circ}$ C. The typical requirement is a creep rupture life of 100,000 hr at 700 $^{\circ}$ C under a stress of 100 MPa.

In nickel based superalloys, the approach has been to strengthen the matrix and to increase the volume fraction of γ' . In addition, the grain boundaries are reinforced by carbide precipitation and by the use of minor addition of boron and zirconium. Conversely, the mechanism of the trace elements on grain boundary reinforcement is not fully studied.

In this study it was observed that the addition of trace elements influenced not only the morphology of grain boundary but also the creep resistance. As a result of the evaluation on microstructure and the creep test we have to clarify the effect of the trace elements.

Keywords: Nickel based superalloy, Boron, Zirconium, Creep resistance, Grain boundary

1. Introduction

It is well known that the grain boundaries (GBs) structure is mainly influenced mechanical properties at elevated temperature such as creep resistance in nickel base superalloys. There are commonly investigated several phase and structure in GB including carbides, gamma prime and so on. The carbides are classified primary MC type carbide and precipitation carbides such as M_6C , $M_{23}C_6$ type [1].

The effect of trace elements is discovered improving the creep rupture time in nickel base superalloy by interaction of melts and crucible and this phenomena is caused that the boron and zirconium are segregated in GBs, and disturbed formation of micro crack and coarsening of carbides in GBs [2]. Many researchers studied the mechanism of trace elements, but it is not clarify about below mentioned.

- 1) the changes of morphology of GBs by addition of trace elements,
- the effects of forming carbides by addition of trace elements
- 3) the effects of creep resistance of GBs morphology

In this study, we want to study the influence of addition of trace element between the creep resistance and microstructure in GBs. So we added trace elements in our designed alloy LESS 1 (Ni - 22Cr - 20Co - 2W - 2Nb - 1.5Ti - 1.5Al - 0.03C), evaluated microstructure in GBs and creep resistance.

2. Experimental procedures

2.1 Materials and processes

The experiments were performed on vacuum induction melting (VIM) LESS 1 alloy having the nominal composition (LESS 1) and addition of trace elements such as boron and zirconium. After the casting, the specimens are conducted homogeneous treatment (1200 \degree C, 16 hr), forging at 1100 \degree C, solution treatment (1200 \degree C, 30 min) and aging treatment (800 \degree C, 16 hr). After the heat treatment and forging, the specimens are prepared for metallography and small punch creep test.

2.2 Metallography

The metallographic specimens were ground using 200-2000 grit abrasive SiC papers. Immediately after the last grinding step, the specimens were washed with water, cleaned ultrasonic in cleaner with ethanol and dried. The fine polishing was conducted using 6 and 1 μ m water based diamond compounds. After the polishing, the specimens were washed, cleaned and dried in the same method as the above mentioned.

The polished specimens were performed on electrical etching by mixture of perchloric acid, propionic acid and ethanol. The etched specimens were evaluated the microstructure on grain boundary using SEM.

2.3 Small punch creep test

The small punch creep test specimen were cut 10 x 10×0.6 mm by wire cutting machine and ground 200 - 2000 grit abrasive SiC papers both sides of the specimen to avoid the influence of heat affected surface during cutting stage. The last grinding step, the size of specimens were $10 \times 10 \times 0.5$ mm.

The prepared specimens were elevated up to 700 $^{\circ}$ C in inert atmosphere using Ar gas, and kept at 700 $^{\circ}$ C for 1 hr because of thermal maintenance. After then it was loaded 50kgf and evaluated the rupture time of specimens by small punch creep test. After creep test we observed the rupture surface by SEM.

3. Results and discussion

The composition of trace element in fabricated specimens are listed in table 1.

In this study, the significant changes by addition of trace elements are morphology of carbides in GBs.

It is observed the similar results that MC and $M_{23}C_6$ carbidse are distributed on GBs in LESS 1 and LESS 1 (Zr) alloy. However in the case of LESS 1 (B), the morphology of GBs is changed that the MC carbides could not be observed, only γ' film with carbides were formed in GBs. This resluts was represented at Fig. 1.

It is clarify that the effect of trace elements is affected to form the carbide, zirconium is stabilized MC carbide and boron is formed γ' film with carbides during aging treatment.

Fig. 2 shows the results of creep test, the creep rupture time is similar between LESS 1 and LESS 1 (Zr), the rupture time of LESS 1 (B) alloy is significantly improved.

After the test we observed the rupture surface by FE-SEM and it is shown at Fig. 3. It is very interested that the surface of LESS 1 and LESS 1 (Zr) alloy is shown traditional fracture of intergranular, the case of LESS 1 (B) is similar observed intergranular fracture but remained precipitation on the surface.

This results show that formation of γ' film with carbides is more effective about creep resistance than MC and M₂₃C₆ carbide in GBs

Table 1 The composition of trace elements (ppm).

Alloy	В	Zr
LESS 1	10	30
LESS 1 (Zr)	10	110
LESS 1 (B)	90	30



Fig. 1 FE-SEM image of the morphology on GBs (a) LESS 1, (b) LESS 1 (Zr) (c) LESS 1 (B)



Fig. 2 The results of small punch creep test



Fig. 3 FE-SEM image of the rupture surface (a) LESS 1 (Zr) (b) LESS 1 (B)

4. Conclusions

The effect of trace elements is summarized below

- 1) the effect of trace elements on GBs is formation of carbide,
- 2) a point of view about creep resistance, it is more effective boron than zirconium because of formation of γ' film with carbides.

References

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