[Article ID] 1003- 6326(2002)01- 0083- 05

Effect of heat treatment on microstructure and high temperature tensile properties of cast nickel-base superalloy with high W, Mo and Nb contents①

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[Abstract] The microstructural features and high temperature tensile properties of M963 superalloy at as-cast, as-solutioned and as-aged conditions were investigated in detail. The results show that the solution treatment at 1 220 ℃ for 4 h, AC causes an increase in high temperature yield strength but a drastic drop in high temperature ductility due to the precipitation of both the secondary carbide M_6C along grain boundaries and at the interdendritic regions and very fine γ′ particles in the dendrite cores. Aging treatment following the solution treatment can improve the high temperature tensile properties of M963 superalloy due to the coarsing of the γ′ precipitate. One stage aging at 850 ℃ for 16 h following the solution treatment causes an increase in both strength and ductility of alloy M963, and two-stage aging of 1 089 ℃/2 h, AC plus 850 ℃/16 h, AC following the solution treatment further increases the ductility of alloy M963 but slightly decreases its strength.

[Key words] cast nickel base superalloy; mechanical properties; microstructure; heat treatment
[CLC number] TG 156.94 **[Document code]** A

1 INTRODUCTION

Ni-base superalloys are widely used in gas turbine components, which require high temperature strength, creep and fatigue resistance, excellent ductility, good impact resistance and adequate resistance to hot corrosion[1]. These superalloys are strengthened through carbide precipitation at the grain boundaries and γ′ precipitation within the grains. Discrete-type grain boundary carbides are generally considered beneficial since they reduce the grain boundary sliding and therefore delay the onset of creep cavitation and rupture[2]. Among the strengthening mechanisms derived by various microstructural features, the strengthening of the γ′ precipitate is the most important one since Ni-base superalloys mainly derive their high temperature creep resistance from the precipitation of an $L1_2$ ordered γ′ phase[3-5]. This phase is usually $Ni_3(Al, Ti)$ coherently in the fcc matrix. The γ′ dispersion is established to hinder the dislocation movement within the grain interiors, and the minimum creep rate has shown to vary as a function of the cube root of the γ′ volume fraction[6,7].

M963 is a cast equiaxed Ni-based superalloy, which has excellent high temperature strength and service temperature capability, mainly due to the addition of a larger amount of the refractory elements such as Mo, W, and Nb[8]. The conventional heat treatment of this alloy is solution treatment at 1 220 ℃ for 4 h followed by air cooling. However, this superalloy treated by the above solution treatment usually has low ductility, especially under the service condition of high temperature.

In this paper, the microstructural features and high temperature tensile properties of M963 superalloy at various conditions, such as as-cast, as-solutioned and as-aged, are investigated in detail. An improvement in both ductility and strength at high temperature has been obtained by aging treatment.

2 EXPERIMENTAL

The M963 superalloy was prepared by vacuum induction melting then cast into round ingots and cut into pieces. The pieces were remelted in a vacuum furnace, and then cast into round bars of 16 mm in diameter and 80 mm in length. The pouring and mold temperatures were 1 500 ℃ and 900 ℃, respectively. The chemical composition of the M963 superalloy is given in Table 1. Parts of the casting bars were subjected to heat treatments as described in Table 2. The standard cylindrical specimens with a gauge length of 25 mm and a gauge diameter of 5 mm were machined longitudinally from the heat-treated bars and the as-

① [Received date] 2001- 04- 28; [Accepted date] 2001- 06- 11

cast bars.

Table 1 Chemical composition of M963 superalloy (mass fraction, %)

C	Cr	Co	W	Mo	Al	Nb
0.13	8.80	9.69	10.68	1.49	5.56	1.10

Ti	S	P	O	N
2.42	0.0009	0.002	0.0007	0.0008

Table 2 Solution and aging heat treatments

Solution treatment	1220 ℃/4h, AC
One-stage aging treatment following solution treatment	1220 ℃/4h, AC+ 850 ℃/16h, AC
Two-stage aging treatment following solution treatment	1220 ℃/4h, AC+ 1089 ℃/2h, AC+ 850 ℃/16h, AC

(AC= air cooling)

The tensile tests were carried out on a DCX-25T high temperature mechanical machine at 900 ℃. The microstructures were examined on ISM-6301F scanning electron microscopy (SEM) equipped with energy disperse spectrometer (EDS) for microanalysis. The volume fractions of the γ' precipitate in the various conditions were determined by area measurement and point counting technique on representative SEM micrographs[9]. The γ' precipitate size measurement was also undertaken on the SEM micrographs.

3 RESULTS

3.1 Microstructure

3.1.1 As-cast

The SEM back-scatter electron image (Fig. 1(a)) shows the typical microstructure of the as-cast M963 superalloy, which contains γ phase, γ' precipitate in cubic shape, a large amount of γ-γ' eutectic and MC carbide. The carbide is mainly distributed in the interdendritic region and has the script-like shape. The EDS analyses demonstrated that the MC carbide has the following composition (mole fraction, %): 1.11Al, 40.32Ti, 3.47Cr, 1.64Co, 13.54Ni, 20.73Nb, 3.98Mo, and 15.21W. It is also clear in Fig. 1(a) that the structure is dendritic. The EDS results showed the dendritic arms are rich in Cr, Co, W, while the interdendritic areas are rich in Al, Ti, Nb. The γ' precipitate particles at the dendrite cores have the average size of 1.0 μm and volume fraction of 40.4% approximately under the as-cast condition.

3.1.2 As-solutioned

Fig. 1(b) shows the microstructure of the alloy after solutioning at 1220 ℃ for 4h followed by air cooling. It can be seen that the γ' phase at the dendrite cores is almost completely solutioned, while some coarse γ' particles at the interdendritic regions remained in addition to the γ-γ' eutectic. Secondary carbide phases with square-like shape are observed along the grain boundaries and at the interdendritic

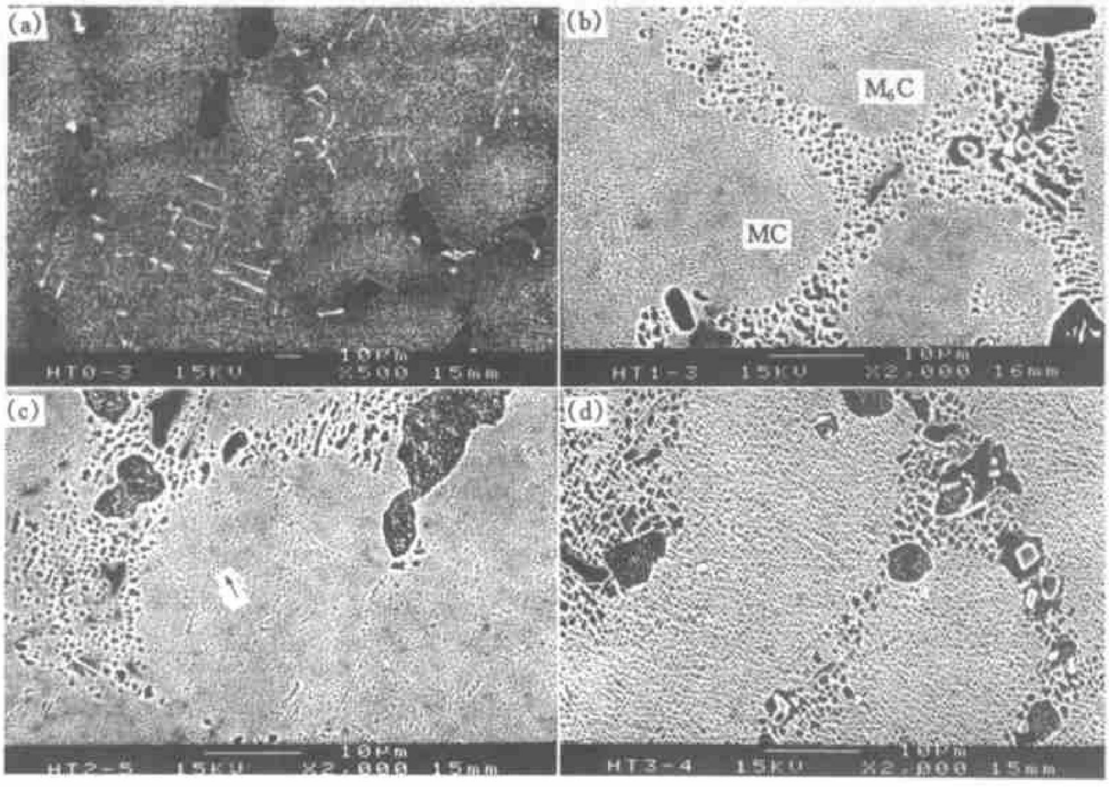

Fig. 1 Microstructures of M963 superalloy treated by various heat treatment
(a) —As-cast, back-scatter electron image; (b) —1220 ℃/4h, AC, secondary electron image (SEI);
(c) —1220 ℃/4h, AC + 850 ℃/16h, AC, SEI; (d) —1220 ℃/4h, AC+ 1089 ℃/2h, AC+ 850 ℃/16h, AC, SEI

regions. The EDS analysis showed that the secondary carbide has the following composition (mole fraction, %): 2.11Al, 2.74Ti, 13.57Cr, 9.55Co, 31.80Ni, 3.0Nb, 6.58Mo, and 30.65W. Since the alloy is rich in W, Mo, the secondary carbide is of the M_6C type, where M is predominantly W, Mo.

At high magnitude, fine cubic γ' precipitates were found in the dendrite cores. It indicates that the fine cubic γ' phase has precipitated in the process of air cooling after solution treatment. The size and volume fraction of the fine γ' precipitate particles are about 0.20 μm and 33.7%, respectively. As compared with the as-cast condition, the γ' precipitate is much finer and a little less, which indicates that the γ' phase has not precipitated completely in the process of air cooling after solution treatment and the γ matrix is supersaturated in Al, Ti, and Nb.

3.1.3 As-aged

Compared with the as-solutioned condition, the aging heat treatment does not significantly change the amount of coarse γ', γ-γ' eutectic and carbides (Fig. 1(c), (d)), but increase the volume percent of fine γ' precipitate at dendrite cores from 33.7% under the as-solutioned condition to 51.9% after aging at 850 ℃ for 16 h, and its size from 0.2 μm to 0.25 μm. When a high temperature aging treatment at 1 089 ℃ for 2 h is added to the heat treatment, the γ' precipitate coarsens greatly whose size reaches about 0.4 μm, but the volume fraction of γ' precipitate does not increase further.

3.2 High temperature tensile properties

The tensile properties of M963 superalloy treated by various heat treatments tested at 900 ℃ are given in Table 3. It can be seen that the yield strength of M963 superalloy at 900 ℃ is increased by solution treatment, but the tensile strength is not, especially the tensile elongation is decreased greatly (from 8.8% to 2.4%). After the M963 superalloy is aged at 850 ℃ for 16 h following the solution treatment, both the tensile strength and elongation of this superalloy are increased obviously. A high temperature aging at 1 089 ℃ for 2 h further increases the tensile elongation, which reaches 6.0%, however, the tensile strength is slightly lowered under this condition. The improvement in ductility at high temperature will be beneficial to the service of the alloy.

Table 3 Tensile results of M963 superalloy treated by various heat treatment at 900 ℃

Heat treatment	σ_b/MPa	$\sigma_{0.2}$/MPa	δ/%
As-cast	812	682	8.8
As-solutioned	812	747	2.4
One-stage aging treatment following solution treatment	928	761	4.0
Two-stage aging treatment following solution treatment	911	753	6.0

3.3 Fractographic observation

In order to investigate the effect of heat treatments on fracture behavior at elevated temperature, both the fracture surface and the longitudinal section adjacent to the fracture region were examined. The fractographs of M963 superalloy experienced various heat-treatments and tensile tested at 900 ℃ are shown in Fig. 2. It can be seen that the heat treatments affect remarkably the fracture behavior of the M963 superalloy. The rupture of the as-cast alloy occurs mainly along the interface of script-like MC carbide (Fig. 2(a)), while the cracks of the as-solutioned alloy initiate at the opened secondary carbide M_6C (Fig. 2(b)). The aged alloys exhibit a relatively ductile fracture surface (Fig. 2(c) and (d).

Fig. 3 and Fig. 4 show micrographs of the longitudinal section of the as-solutioned and as-aged M963 superalloys tensile tested at 900 ℃. It is evident that a crack initiates at the interface between the matrix and secondary carbide M_6C under the as-solutioned condition (Fig. 3). The as-aged alloy, however, allows much slide deformation without causing crack at the interface between matrix and carbide (Fig. 4).

4 DISCUSSION

It has been reported that in other cast equiaxed Ni-base superalloys the yield strength was increased and tensile elongation was decreased remarkably by solution treatment[10]. HUANG et al[10] proposed that the low ductility of the as-solutioned alloy is due to fine γ' precipitate hardening within the grain interior which make it difficult to coordinate the deformations of the adjacent grains. Previous investigation[11] indicated that the low ductility of the as-solutioned M963 superalloy is related to the precipitation of the square-like secondary carbide M_6C in the process of solution treatment. According to this investigation, the following mechanism can be drawn. Because the alloy M963 contains a large amount of W, Mo and Nb, which are all considered as the most effective solid solution strengthening elements[12], and the as-solutioned alloy M963 has very fine γ' particles at the dendrite cores, which cause greatly precipitation hardening, the deformation of the alloy within the dendrite cores is very difficult and thus the stress concentration at the interface between matrix and the secondary carbide M_6C located at grain boundaries and interdendritic regions is high enough to make the M_6C carbide particles break (Fig. 2(b)) or to initiate cracks along the interface (Fig. 3). Therefore, the fracture of the alloy M963 under the as-solutioned

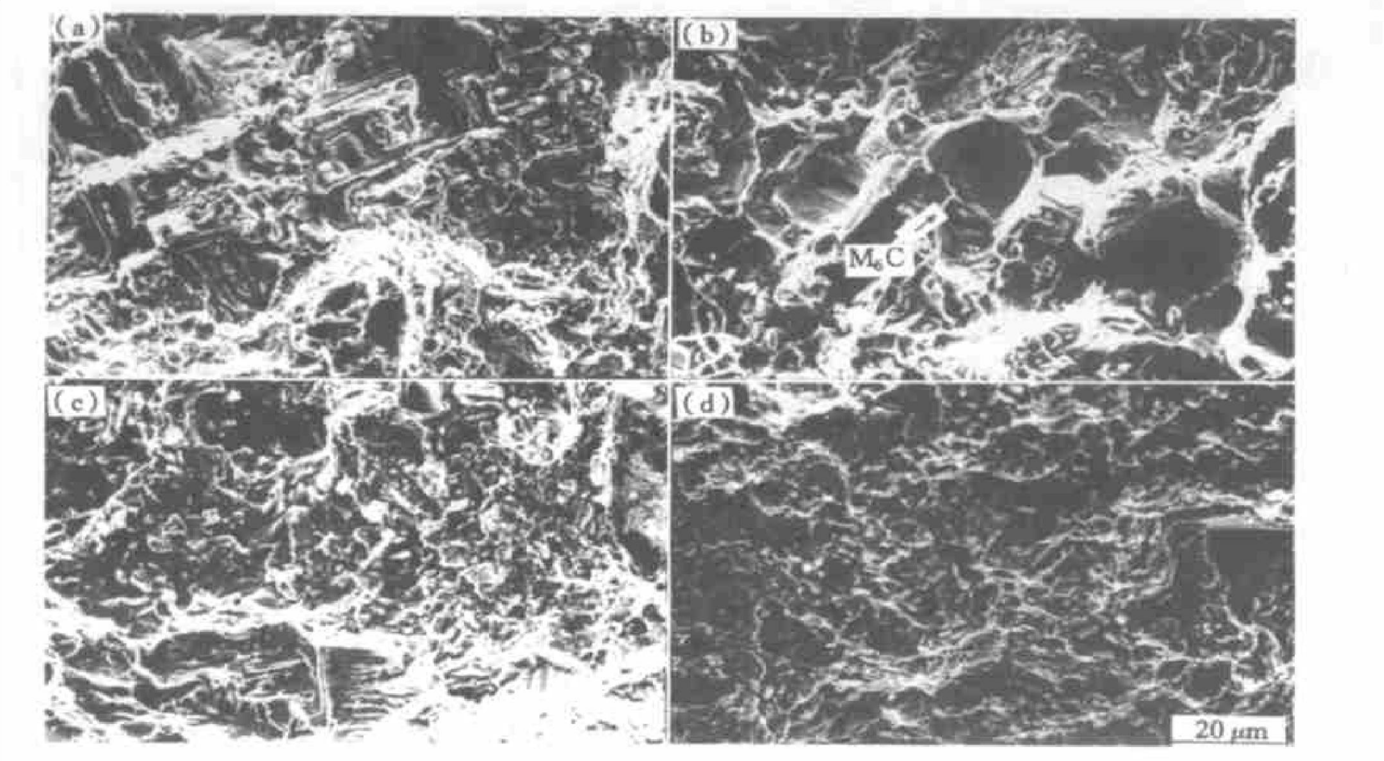

Fig. 2 Fractographs of M963 superalloys at various heat-treated conditions tensioned at 900 ℃
(a) —As-cast; (b) —As-solutioned; (c) —One-stage aged; (d) —Two-stage aged

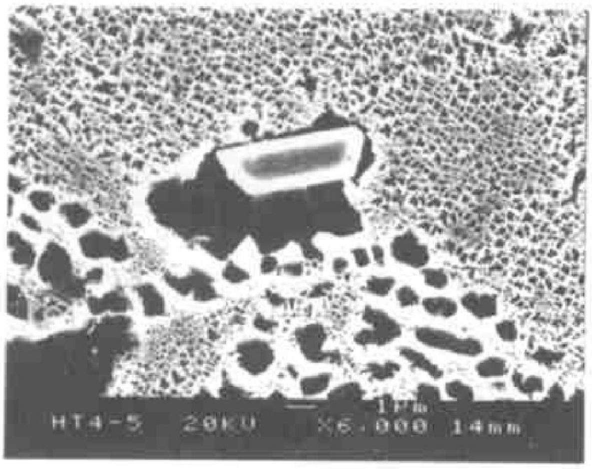

Fig. 3 Micrograph of longitudinal section of as-solutioned M963 superalloy tensioned at 900 ℃

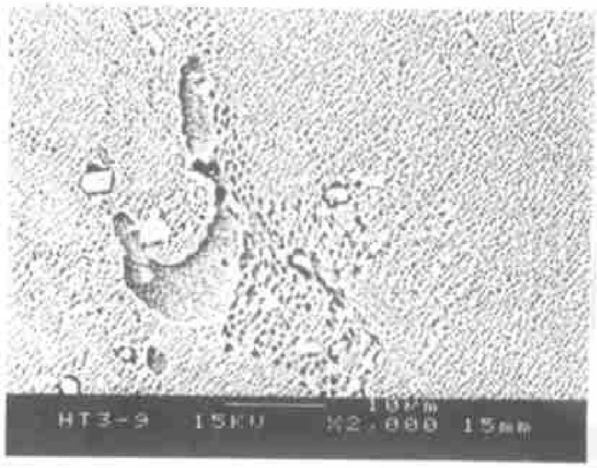

Fig. 4 Micrograph of longitudinal section of M963 superalloy with two-stage aging tensioned at 900 ℃

condition occurs without much plastic deformation.

When the alloy M963 is aged at 850 ℃ for 16 h, the fine γ' precipitate grows up, thus the deformation in matrix becomes easier and the stress concentration around the M_6C carbide particles lower. Therefore, the initiation of cracks is delayed and both the strength and ductility are increased. Because the aging temperature of 850 ℃ is not high and the growth rate of γ' precipitate is low, the aging treatment at 850 ℃ for 16 h causes only a little increase in ductility. When a high temperature aging treatment at 1 089 ℃ for 2 h is used, the size of γ' precipitate increases obviously, as a result that the ductility of the alloy is improved remarkably and the strength, in the meantime, is decreased. The fracture surface morphology of the alloy under the two-stage ageing conditions of 1 089 ℃/2 h, AC plus 850 ℃/16 h, AC following the solution treatment at 1 220 ℃/4 h, AC tensile tested at 900 ℃ exhibits an obvious ductile fracture feature (Fig. 4).

5 CONCLUSIONS

1) The solution treatment at 1 220 ℃ for 4 h, AC causes an increase in high temperature yield strength but a great decrease in high temperature ductility due to the precipitation of the secondary carbide M_6C along grain boundaries and at the interdendritic regions and very fine γ' particles in the dendrite

cores.

2) Aging treatment following the solution treatment can improve the high temperature tensile properties of M963 superalloy due to the coarsening of the γ′ precipitate. One-stage aging at 850 ℃ for 16 h following the solution treatment causes an increase in both strength and ductility of alloy M963, and two-stage aging of 1089 ℃/ 2 h, AC plus 850 ℃/ 16 h, AC following the solution treatment further increases the ductility of alloy M963 but slightly decreases its strength.

[REFERENCES]

[1] Sims C T, Stoloff N S, Hagel W C, et al. Superalloy II [M]. New York: John Wiley & Sons, Inc, 1987. 24.

[2] Baldan A. Effects of grain size and carbides on the creep resistance and rupture properties of a conventionally cast nickel-base superalloy [J]. Z Metallkd, 1992, 83(10): 750- 757.

[3] Baldan A. The Effect of initial γ′ morphology on the high temperature creep resistance of a nickel-based superalloy [J]. Z Metallkd, 1992, 83(5): 324- 330.

[4] Balikci E, Raman A, Mirshams. Influence of various heat treatments on the microstructure of polycrystalline IN738LC [J]. Metall Mater Trans A, 1997, 28A(10): 1993- 2003.

[5] Jensen R R, Tien J K. Temperature and strain rate dependence of stress-strain behavior in a nickel-base superalloy [J]. Metall Trans A, 1985, 16A(6): 1049- 1068.

[6] Hopkins D E, Gibbons T B. Influences of some structural factors on the creep strength of wrought precipitation-hardened Ni-Cr alloys [A]. Proceedings of a Meeting on Creep Strength in Steel and High-temperature Alloys [C]. Organized by the Iron and Steel Institute Held at the University of Sheffield, 1972. 165.

[7] Jackson J J, Donachie M J, Henricks R J, et al. The effect of volume percent of fine γ′ on creep in DS Mar-M200+ Hf [J]. Metall Trans A, 1977, 8A(10): 1615 - 1620.

[8] YUAN Chao, SUN Xiao-feng, YIN Feng-shi, et al. Characteristics of hight temperature rupture of a cast nickel base superalloy M963 [J]. J Mater Sci Technol, 2001, 17(3): 1- 4.

[9] CAI Yu-lin, ZHENG Yun-rong. Investigation on Microstructures of Superalloy [M], (in Chinese). Beijing: National Defensive Industrial Press, 1986. 70- 77.

[10] HUANG Qian-yao, LI Han-kang. Superalloy [M], (in Chinese). Beijing: Metallurgical Industry Press, 2000. 143.

[11] YIN Feng-shi, YUAN Chao, SUN Xiao-feng, et al. An investigation on the microstructure of a cast nickel-base superalloy M963 [J]. Heat Treatment of Metals, (in Chinese), 2001, 26(7): 1- 2.

[12] CHEN Guo-liang. Superalloy [M], (in Chinese). Beijing: Metallurgical Industry Press, 1988. 167- 177.

(Edited by YUAN Sai-qian)