THE INFLUENCE OF δ PHASE ON MECHANICAL PROPERTIES OF ATI 718PLUS ALLOY

Minqing Wang^{1,2,3}, Qun Deng^{1,3}, Jinhui Du^{1,3}, Zhiling Tian¹, Jing Zhu²

1. Central iron and steel research institute, Beijing 100081, P.R. China
2. School of Materials Science and Engineering, Tsinghua University, Beijing, 100084, P.R. China
3. Beijing Key Laboratory of advanced high temperature materials, Beijing 100081, P.R. China

Abstract

The relation between the amount of δ phase and mechanical properties in ATI 718Plus alloy has been explored for different heat treatment systems in this paper. The results showed that the stress rupture life of ATI 718Plus prolonged significantly and the tensile strength also improved slightly with solution temperature increase. Microstructures were analyzed by field emission scanning electron microscope (FESEM) and quantitative determination of phases for different heat treatment systems was further conducted by electrolytic phase isolation and followed by micro-chemical and XRD analyses. At last, the favorable heat treatment system in ATI 718Plus was suggested in this paper.

Key words: ATI 718Plus alloy, δ phase, stress rupture life, tensile property.

Introduction

ATI 718Plus (hereafter referred to as 718Plus) is a newly developed nickel-based superalloy by ATI Allvac designed to replace Inconel 718 at higher temperature in static and rotating applications in gas turbine engines. The temperature capability is at least 55°C higher than that of alloy Inconel 718 alloy and effectively fills the longstanding gap between the two most widely used wrought superalloys, Inconel 718 and Waspaloy^[1,2].

As we know, it is important for δ precipitation in Inconel 718 to adjust mechanical properties. Although the precise effect of δ phase can vary considerably and appears to depend strongly on the location and morphology of the phase^[2-6], it is generally accepted that a small amount of δ precipitation is essential for grain boundary pinning, and that excessive levels of coarse δ are detrimental to properties due to solute depletion of γ'' phase. The precipitation of δ phase can lead to serration of grain boundaries in Inconel 718 and reports suggest that such a microstructure is likely to improve intergranular fatigue crack resistance^[2,7,8]. But it was found by Xie et.al^[9] and Pickering et.al^[3] that the crystal structure and chemistry of δ phase in 718Plus alloy was different from the orthorhombic δ -Ni₃Nb reported previously in Inconel 718. The true structure of δ phase in 718Plus alloy was consistent with hexagonal η -Ni₃Ti structure, but its chemistry was close to Ni₃Al_{0.5}Nb_{0.5}. Because of the different structure and chemistry of δ -phase in 718Plus from δ -phase in Inconel 718, the precipitation and the

Corresponding author. E-mail address: minqingw@yahoo.com

influence on mechanical properties for δ phase in 718Plus was needed to be investigated further.

In this paper, different amounts of δ and γ' in 718Plus were obtained by three different heat treatment systems, and quantitative determination of phases was further conducted by electrolytic phase isolation and followed by micro-chemical and XRD analyses. The tensile property and stress rupture life were also tested. At last, the relationship between mechanical properties and the amount of phases was discussed.

Experiments

Materials

Table I Chemical composition of the experimental alloy

C	P	Cr	Ni	Mo	Nb	Ti	Al	Co	Fe	W	B
0.044	0.012	18.98	BAL	2.78	5.55	0.75	1.56	8.99	9.28	1.07	0.0062

Experimental materials were prepared by vacuum induction melting (VIM) and every ingot is about 50kg. The ingot was forged to $\Phi 100$ mm bars and further refined by vacuum arc remelting (VAR). Then the ingot was rolled to $\Phi 18$ mm bars after homogenizing heat treatment. The chemical composition of experimental alloy is shown in table I. The as-rolled microstructure consists of about ASTM 11 (Fig.1(a)) with little δ phase (Fig.1(b)).

Fig.1 The microstructure of as-rolled samples

Heat treatments

Then the sample was subject to different heat treatments, and the heat treatments details were outlined in Table II. Before solution and aging heat treatment, all the experimental samples were also “pre-solution” heat treated at 870°C for 16h. The solution temperature was gradually increased from 945°C to 975°C.

Table II 718Plus details of heat treatment

Label	Heat treatment details
AR(as-rolled)	No heat treatment, examined in as-received condition
Heat treatment A	870°C × 16h AC + 945°C × 1h AC + 788°C × 8h, 56°C/h furnace cooling to 704°C × 8h AC
Heat treatment B	870°C × 16h AC + 960°C × 1h AC + 788°C × 8h, 56°C/h furnace cooling to 704°C × 8h AC
Heat treatment C	870°C × 16h AC + 975°C × 1h AC + 788°C × 8h, 56°C/h furnace cooling to 704°C × 8h AC

Results

Microstructures

The distribution of δ phase in material A, B and C subjected to different heat treatment was illustrated in Fig.2(a), (b) and (c) respectively. The quantity of δ phase in material A was obviously more than other heat treatment materials. With rising solution temperature, the amount of δ precipitation reduced gradually. It can be seen that the δ precipitation in materials B and C primarily distributes along grain boundary with lath-shaped morphology. In comparing the size of δ phase, it looks larger for low solution temperature specimen than higher solution heat treated specimens.

Fig.2 Microstructures of samples supplied by different heat treatment

Detailed microstructure of field emission scanning electron microscope, SUPRA 55, was used for imaging. It was illustrated in Fig.3(a) that a great deal of γ' and δ phase can precipitate after solution heat treatment, and γ' phase is depleted around δ phase. This is considered to be beneficial to plastic deformation of grain boundary. After aging heat treatment, the smaller size of γ' shown in Fig.3(b) and (c) continued to precipitate. So during solution heat treatment and air cooling, large size γ' and δ phases can precipitate, then more smaller size γ' precipitates during aging heat treatment.

Fig.3 The distribution of γ' and δ phases after heat treatment

Properties

The 704°C tensile properties for 718Plus by different heat treatment were illustrated in table III and every experimental data point was the average value of three experimental data. It can be seen that 718Plus alloy shows excellent tensile property at 704°C, and the ultimate and yield strengths improved slightly with solution temperature increase.

Table III Tensile properties of different heat treatment materials

Label	Temperature (°C)	σ_b /MPa	$\sigma_{0.2}$ /MPa	δ /%	ψ /%
Heat treatment A	704	1140	975	37.0	65.0
Heat treatment B	704	1155	995	37.0	64.0
Heat treatment C	704	1180	1000	32.0	52.0

The stress rupture life of the materials subject to different heat treatment was listed at table IV. It can be seen that the stress rupture life of 718Plus alloy was obviously influenced by solution system. With rising of solution temperature, the stress rupture life at 704°C/621MPa improved from 28.4h to 73.7h for samples A, B and C.

Table IV The stress rupture life of different heat treatment materials

Label	Temperature (°C)	Stress (Mpa)	Average Rupture life (h)	Average Rupture Elongation (%)
A	704	621	28.4	33.5
B	704	621	40.1	37.0
C	704	621	73.7	41.5

Time-Temperature-Transformation diagram

Fig.4 shows the TTT curve for 718Plus calculated using JMatPro 6.0 program. The nose of the TTT curve for γ' precipitation occurs at about 920°C for less than 0.1 h and the nose of the TTT curve for δ precipitation occurs at about 950°C for less than 1h. In comparing the phase formation curves in alloy 718Plus with conventional alloy 718 by Xie^[12], the γ'/γ'' and δ solvus for alloy 718Plus are much higher than that for conventional alloy 718, and is consistent with precipitation hardening phases that are stable to a higher temperature than in alloy 718.

Fig.4 Time-Temperature-Transformation Curves for 718Plus

Phase identification

Quantitative determination of phases for different heat treatment and as-rolled condition materials were conducted by electrolytic phase isolation, followed by micro-chemical analyses. The quantity and chemical composition of δ and γ' phases in different heat treatment specimens were summarized in table V. After pre-solution and low temperature solution (heat treatment A), about 7.3% δ phase precipitated and the quantity of δ precipitation decreased gradually with increasing solution temperature (heat treatment B and C). The as-rolled condition material also included about 22wt.% γ' phase. The solvus temperature of γ' in 718Plus is about 963 °C according to DTA result^[10] and the rolled temperature was as high as 1120°C. So there should be little γ' in as-rolled material and a large number of γ' phase must precipitate during air cooling.

The chemical compositions of δ and γ' phases subject to different heat treatment were almost the same except the slight change for the amount of Cr, Nb, Al, Ti. The quantity of Cr increased in δ -phase with rising solution temperature, and it was opposite for Cr in γ' phase. The quantity of Nb and Ti all increased with rising solution temperature whether in γ' or δ phase. The content of Al had little change in γ' phase, but a slight decrease in δ phase.

Table V Chemical composition of phases in different heat treatment systems

δ phase										
Label	Weight percent	Ni	Co	Fe	Cr	Al	Mo	Nb	Ti	W
AR	0.974	66.55	5.71	1.81	1.86	5.89	0.36	13.14	4.54	0.15
A	7.342	67.47	5.48	1.42	1.25	7.68	0.25	12.52	3.77	0.15
B	5.435	67.36	5.51	1.45	1.36	7.09	0.27	12.92	3.89	0.15
C	2.975	67.15	5.57	1.51	1.54	6.86	0.29	12.95	3.99	0.15
γ' phase										
Label	Weight percent	Ni	Co	Fe	Cr	Al	Mo	Nb	Ti	W
AR	22.398	67.65	3.70	1.68	3.94	11.31	0.32	8.33	2.88	0.20
A	20.450	68.67	2.95	1.39	3.98	11.51	0.33	8.55	2.40	0.22
B	21.774	68.82	2.97	1.35	3.73	11.55	0.31	8.61	2.47	0.21
C	24.051	68.97	2.98	1.34	3.42	11.41	0.28	8.81	2.60	0.19

The experimental result confirmed that the true structure of δ phase in 718Plus alloy was consistent with hexagonal η -Ni₃Ti structure, but its chemistry was close to Ni₃Al_{0.5}Nb_{0.5}, as reported by Xie et al.^[9] Pickering^[3] also considered that the δ phase in 718Plus alloy was consistent with η -Ni₃Ti, but the chemistry was close to Ni₆AlNb with partial ordering of Al and Nb over the prototype Ti sites. Actually, Ni₃Al_{0.5}Nb_{0.5} and Ni₆AlNb are the same from chemical composition, but Ni₆AlNb may be accurate from structure, because the structure of Ni₃Al_{0.5}Nb_{0.5} first reported in the 1960s by Giese^[11] had the Al and Nb atoms randomly distributed over the Ti sites of the Ni₃Ti crystal structure, and the experimental results show that Nb, Ti and Al atoms are ordered over the two distinct Ti sites of the η -Ni₃Ti prototype structure^[3]. We can call this new phase, η -Ni₃Al_{0.5}Nb_{0.5} or η -Ni₆AlNb as δ' -phase which has η -Ni₃Ti structure, but like δ -Ni₃Nb chemistry, to distinguish from true η -Ni₃Ti and δ -Ni₃Nb phases.

Fig.5 γ' phase size distribution by different heat treatment

The statistics of the size distribution of γ' phase in three heat treatment examples was also analyzed. It can be seen from Fig.5 that the size of γ' is not continuous distribution. The majority of γ' distributes at 10~36nm and another mainly distributes greater than 60nm. The experimental results illustrated in Fig.3 indicated that the γ' greater than 60nm precipitates primarily before aging heat treatment, and this can be regarded as primary γ' . After aging heat treatment, a large number of 10~36nm γ' can precipitate, and this part of γ' can be regarded as secondary γ' . With rising of solution temperature, the amount of 10~36nm γ' in total γ' increases significantly from 77.1% to 93.2% and the amount of 140~300nm γ' decreases from 15.0% to 5.6%, accordingly.

Discussion

Although δ' precipitation in 718Plus has different crystal structure with δ in Inconel 718 alloy, its morphology and chemical composition indicate that it should have similar effect. Because of its morphology, the δ' -phase does not contribute significantly to the hardening of the alloy. On the contrary, its presence implies a loss of hardenability due to the depletion of γ' (Fig.3). A moderate fraction of δ' is effective in limiting grain growth during solution treatment and the appropriate morphology has been shown to provide resistance to grain boundary creep fracture^[13,14]. It can restrict the slip of grain boundary and cracking path, which is beneficial to relieve stress concentration and eliminate notch sensitivity. The beneficial effect was also observed by Pickering et.al^[3] that δ' phase influenced the morphology of grain boundaries, such that the latter appeared distorted or serrated. However, if excessive level of δ' exists at grain boundary, total γ' content decreases and the strength of matrix reduces accordingly.

All the specimens were subjected to 870°C × 16h AC + 945°C, 960°C or 975°C × 1h AC + 788°C × 8h, 56°C/h furnace cooling to 704°C × 8h AC heat treatment. From TTT curve for 718Plus in Fig.5, the nose of the TTT curve for δ' precipitation occurs at about 950°C and δ can precipitate at 870°C less than 1h. So a large number of δ can precipitate after pre-solution at 870°C for 16h. Then after solution heat treatment above 945°C, some amount of δ will dissolve to matrix and the equilibrium quantity decreases gradually with rising solution temperature. While the nose of the TTT curve for γ' precipitation occurs at about 920°C and γ' can precipitate at 870°C less than 0.1h, the primary γ' can precipitate and further coarsen

during long time pre-solution heat treatment at 870°C for 16h. During solution heat treatment for 1h and air cooling, γ' can also coarsen further. From distribution of δ and γ' illustrated in Fig.3, it also clearly indicated that the primary γ' precipitated before aging heat treatment and the majority of γ' , i.e. the secondary γ' precipitated during aging.

From table V, the total weight percent of δ' and γ' phase was almost constant between 27% and 28wt.%, and the increase of one phase was always accompanied with the decrease of the other. With rising solution temperature, the quantity of δ' phase reduces from 7.3% to 3.0% and that of γ' phase increases from 20% to 24%. Accordingly, the tensile strength improved slightly, but the stress rupture life prolonged significantly from 28.4h to 73.7h (Table IV). The improvement of tensile strength is mainly related to the increase of total γ' phase content, especially the increase of the secondary γ' . From the contrastive analysis of stress rupture life and phases constitute, it clearly can be seen that the stress rupture life was prolonged. So it can be deduced that favorable stress rupture life can be obtained under the condition that δ' phase was as little as possible through heat treatment. But it is dangerous if the amount of δ' phase is too little at grain boundary and notch sensitivity can take place. So, after pre-solution heat treatment, higher solution temperature was more favorable to tensile and stress rupture life properties improvement for 718Plus under the condition that the δ' phase was not too little to cause notch sensitivity. The experimental results showed that the favorable quantity of δ' phase was not more than 2.9wt.%. The best amount of δ' phase is requires further investigation.

Conclusions

1. Experimental results showed that higher solution temperature was beneficial to tensile and stress rupture life after pre-solution heat treatment for 718Plus alloy under the condition that the δ' phase was sufficient to prevent notch sensitivity.
2. The primary γ' precipitates during solution heat treatment and air cooling, and the secondary γ' precipitates during aging heat treatment.
3. True δ phase in 718Plus was confirmed as η - $\text{Ni}_3\text{Al}_{0.5}\text{Nb}_{0.5}$, which has η - Ni_3Ti structure, but $\text{Ni}_3\text{Al}_{0.5}\text{Nb}_{0.5}$ chemistry, and this new phase was suggested to be called δ' -phase to distinguish from the true η - Ni_3Ti and δ - Ni_3Nb phases.

Acknowledgements

This work was supported by the National Basic Research Program of China (No.2010CB631203) .

Reference

- [1] R.L. Kennedy, W.D. Cao, T.D. Bayha, and R.A. Jeniski, “Developments in Wrought Nb Containing Superalloys (718+100°F),” Niobium for High Temperature Applications, ed. by Young-Won Kim and Tadeu Carneiro, TMS, 2004, 11-2
- [2] R.L. Kennedy, “Allvac 718Plus, superalloy for the next forty years,” Superalloys 718,625, 706 and derivatives. Edited by E.A.Loria, PA:TMS. 2005,1-14.
- [3] E.J. Pickering et.al., “Grain-boundary precipitation in Allvac 718Plus,” *Acta Materialia*, 60 (2012) ,2757-2769.
- [4] Liu Xingbo et al., “Effect of thermal-mechanical treatment on the fatigue crack propagation behavior of newly developd Allvac 718Plus alloy,” Superalloys 718 and derivatives. Warrendale, PA:TMS, (2005), 233-242.
- [5] Hayes R, Thompson E, Johnson K, Aichlmayr A, “Effect of heat treatment on the combination stress-rupture properties of Allvac 718plus™,” *Mater. Sci. Eng. A*, 510-511(2009), 256-261.
- [6] Li S et al., “The effect of δ -phase on crack propagation under creep and fatigue conditions in alloy 718,” Superalloys 718 and derivatives, Warrendale, PA:TMS, 1994, 545-555.
- [7] Viskari L, Cao Y, Norell M, Sjöberg G, Stiller K, “Grain boundary microstructure and fatigue crack growth in Allvac 718Plus superalloy,” *Mater. Sci. Eng. A*, 528(2011),2570–80.
- [8] Koul AK, Au P, Bellinger N, Thamburaj R, Wallace W, Immariageon JP, “Development of a damage tolerant microstructure for Inconel 718 turbine disc material,” Superalloys 1988, Warrendale, PA: TMS, 1988, 3–12.
- [9] Xishan Xie et al., “Structure Stability Study on a Newly Developed Nickel-Base Superalloy—Allvac® 718Plus™,” Superalloys 718, 625, 706 & Various Derivatives, ed. by E.A. Loria, TMS, 2005,179-191.
- [10] Wei-Di Cao, “Solidification and solid state phase transformation of Allvac® 718Plus™ alloy,” Superalloys 718, 625, 706 & Various Derivatives, ed. by E.A. Loria, TMS, 2005, 165-177.
- [11] Giessen BC, Grant NJ, “New intermediate phases in transition metal systems,” *III Acta Cryst*, 18(1965), 1080.
- [12] Xishan Xie, Chunmei Xu, Gailian Wang, “TTT diagram of newly developed nickel-base superalloy-ALLVAC® 718PLUS™,” Superalloys 718,625, 706 and derivatives. Edited by E.A.Loria, PA:TMS, 2005,193-202.
- [13] Muller JF, Donachie MJ, Superalloys a technical guide. Materials Park, OH: ASM international, 1975, 211–86.
- [14] Sjöberg G, Ingsten NG, “Superalloy 718—metallurgy and applications,” Warrendale, PA: Minerals, Metals & Materials Society, 1991, 603– 621.