Effect of Heat Treatment on the Microstructure and Mechanical Properties of the Modified 718 Alloy

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Abstract

M718 alloy with an extra high Mo content of 7.50 wt% which reduced Nb addition and increased Al and Ti additions within the composition specifications of 718 alloy has been designed to increase the service temperature of 718 alloy. And the effect of the heat treatment on the microstructure and mechanical properties of M718 alloy has been investigated in this study. The results showed that Laves phase precipitated on the grain boundaries of M718 alloy instead of δ -Ni₃Nb phase in 718 alloy, and γ'' and γ' phases precipitated in the matrix of M718 alloy as that in 718 alloy. Increasing the solution temperature from 960 to 1050 °C noticeably reduced the intergranular precipitation of Laves phase. The precipitation of Laves phase was appropriate at 1020 °C for improving the grain boundary cohesion. Increasing the two-stage aging temperatures markedly increased the sizes of γ'' and γ' phases. As a result, the strength of M718 alloy increased.

Keywords Heat treatment · Mo · 718 alloy · Microstructure · Mechanical properties

1 Introduction

718 alloy is a precipitation-hardened Ni–Fe base superalloy, which is strengthened mainly by γ'' -Ni₃Nb phase which has the DO₂₂ structure and partially by γ' -Ni₃(Al, Ti) phase which has the L1₂ structure [1–3]. Due to its excellent comprehensive mechanical properties, oxidation and hot corrosion resistance, good processability and weldability, 718 alloy has been widely applied in aerospace, nuclear power and oil industries [4–6]. However, the long-term service temperature of 718 alloy cannot be higher than 650 °C, because γ'' phase will be instable and then transform into δ phase, and thus lose the strength markedly [7, 8].

In the past 30 years, many researchers have carried out a lot of work to improve the microstructure stability of 718 alloy and intended to develop a new 718 type alloy used beyond 650 °C [9]. One of the methods is to control the precipitation behavior of γ'' phase and γ' phase by means of modifying Al, Ti and Nb contents, and the precipitates with a sandwich structure of γ'' phase and γ' phase can be obtained by appropriate heat treatment and hence the mechanical properties beyond 650 °C can be improved. Additionally, the proper addition of P and B can lead to a significant improvement in stress rupture life [10–14]. Some researchers have strengthened the alloy matrix by adding solid solution elements. As an important solid solution element, Mo would reduce the bulk diffusion rate and retard the coarsening of γ'' phase and γ' phase of 718 alloy [15, 16], and the increase in Mo content could have a significant effect on the creep rate [17–20].

The present paper concentrates on the effect of the heat treatment on the microstructure and mechanical properties of the modified 718 alloy. For comparison, a commercial 718 alloy treated by the standard heat treatment was also tested.

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2 Experimental

In order to improve the service temperature of 718 alloy, a modified 718 alloy (namely M718) was designed by additions of 0.70 wt% Al, 1.10 wt% Ti, 4.80 wt% Nb and 7.50 wt% Mo. M718 alloy and commercial 718 alloy were smelted by vacuum induction melting in this study, and their chemical compositions are listed in Table 1. The tested alloys were homogenized and subsequently forged into bars with 30 mm in square section, finally hot-rolled into round bars of 18 mm diameter.

As listed in Table 2, M718 alloy was subjected to four heat treatments as HT1 to HT4, while 718 alloy was only treated by the standard heat treatment HT1. Then, the tensile properties at room temperature and 680 °C and the stress rupture properties at 680 °C and 725 MPa were measured.

After grinded and polished mechanically, the samples for microstructure analysis were etched for 75 s in a solution of 5 g CuCl₂, 100 mL HCl and 100 mL C₂H₅OH. The microstructural evolution and precipitation of the phases were carefully characterized by optical microscopy (OM), field emission gun scanning electron microscopy (SEM, JEOL 6340) and transmission electron microscopy (TEM). Energy-dispersive spectroscopy (EDS) was employed to identify the composition of the phases. The TEM specimens were prepared by mechanically grinding to 50 µm thick and then twin-jet electropolishing in a solution of 90% C₂H₅OH and 10% HClO₄ at − 20 °C under 20 V.

3 Results

3.1 Microstructures

As shown in Fig. 1, the microstructures of M718 alloy and 718 alloy were all homogeneous after different heat treatments. The grain size of M718 alloy was about 11 µm (as

shown in Fig. 1a–c), and that of 718 alloy was about 4.5 µm (as shown in Fig. 1d). The white particles with a diameter of approximately 1 µm uniformly precipitated on the grain boundaries in M718 alloy, as presented in Fig. 2a–c. The number of the particles on the grain boundaries decreased noticeably when the solution temperature increased from 960 to 1020 °C, and reduced slightly when the solution temperature increased from 1020 to 1050 °C. When 718 alloy was subjected to HT1, as shown in Fig. 2d, a large number of rod-like δ phases precipitated on the grain boundaries.

Using the SEM/EDS microanalysis, it was found that the white particles on the grain boundaries of M718 alloy were rich in Mo and Nb, as presented in Fig. 3. It was identified to be Laves phases by selected area electron diffraction (SAED), as shown in Fig. 4. However, no δ phases were observed in M718 alloy. Collier et al. [21] found that the driving force for the formation of δ phases decreased with increasing (Al + Ti) content and reducing the Nb content in 718 alloy. The lower content of Nb and the addition of Mo in M718 alloy resulted in the precipitation of Laves phase which in turn consumed a large number of Nb. Therefore, δ phases did not precipitate on the grain boundaries. In addition, Mo was believed to segregate to the grain boundaries in superalloys [22–28]. Thus, the concentration of Mo at the grain boundaries of M718 alloy was higher than that of 718 alloy. During the nucleation and growth of δ phase, Mo was expelled out of δ phase and excessive Mo was gathered at the grain boundaries, which in turn inhibited the precipitation of δ phase.

The morphologies of the strengthening phases are shown in Fig. 5a–c, and the SAED pattern from the [0 0 1] matrix zone axis of M718 alloy is shown in Fig. 5d. The $\langle 0 0 1 \rangle$ and $\langle 0 1 1 \rangle$ type superlattice reflections are from both γ'' phase and γ' phase, while the $\langle \frac{1}{2} 1 0 \rangle$ reflections can only be associated with γ'' phase [29]. As shown in Fig. 5a, b, the morphologies of γ'' phase and γ' phase in M718 alloy were similar with those in 718 alloy, showing dish shaped and spherical shaped, respectively. In addition,

Table 1 Chemical composition of the tested alloys (wt%)

Alloy	C	Ni	Cr	Al	Ti	Nb	Mo	P	B	Fe
M718	0.04	53.0	19.0	0.70	1.10	4.80	7.50	0.002	0.004	Bal.
718	0.04	53.0	19.0	0.50	1.00	5.30	3.10	0.002	0.004	Bal.

Table 2 Heat treatment preformed on the tested alloys

HT No.	Solution	Aging
1	960 °C × 1 h, AC	720 °C × 8 h, cooled at 55 °C/h to 620 °C, 620 °C × 8 h, AC
2	960 °C × 1 h, AC	750 °C × 8 h, cooled at 55 °C/h to 620 °C, 620 °C × 8 h, AC
3	1020 °C × 1 h, AC	750 °C × 8 h, cooled at 55 °C/h to 620 °C, 620 °C × 8 h, AC
4	1050 °C × 1 h, AC	750 °C × 8 h, cooled at 55 °C/h to 620 °C, 620 °C × 8 h, AC

Fig. 1 OM micrographs of M718 alloy treated by **a** HT1, **b** HT3, **c** HT4, **d** 718 alloy treated by HT1

the typical combined morphology of γ'' phase and γ' phase was obtained. When the aging temperatures increased from 720 to 750 °C and from 620 to 650 °C, the average diameter of γ'' phase increased from 11 to 20 nm, and that of γ' phase increased from 10 to 18 nm. After the standard heat treatment, the average diameters of γ'' phase and γ' phase were, respectively, 17 and 13 nm in 718 alloy. This result was consistent with the previous study [30] that the diameters of γ'' phase and γ' phase were < 20 nm. Compared with that of 718 alloy, Mo decreased the size of γ'' phase after the standard heat treatment. Many researchers [31–33] studied the coarsening behavior of γ'' phase and showed that the coarsening kinetic of γ'' phase followed the time-law prediction of the LSW theory of Nb diffusion-controlled growth. Mo decreased the diffusion rate of Nb in the matrix, and thus inhibited the growth of γ'' phase. It was found that larger γ'' phase was easier to transform into δ phase [34–37]. Therefore, Mo inhibited the transformation from γ'' phase to δ phase by decreasing the diffusion rate of Nb.

3.2 Tensile Properties

The tensile properties of the tested alloys at room temperature and 680 °C are listed in Table 3. When the solution temperature increased from 960 to 1020 °C and aging

temperatures increased from 720 and 620 to 750 and 650 °C (from HT1 to HT3), both the strength and ductility of M718 alloy at room temperature and 680 °C increased noticeably. When the solution temperature increased to 1050 °C (HT4), the tensile strength was not improved further at room temperature, but began to decrease at 680 °C. It should be noticed that the heat treatment HT3 was optimum for M718 alloy to obtain the best tensile properties both at room temperature and 680 °C in the present study. The yield strength of M718 alloy was lower than that of 718 alloy, while the ultimate strength and ductility were comparable with those of 718 alloy.

Figures 6 and 7 show the fractographs of the tested alloys tensiled at room temperature and 680 °C. The fracture surfaces of M718 alloy showed the typical intergranular fracture both at room temperature and 680 °C, as shown in Figs. 6, 7a, and the secondary intergranular cracks were observed on the fracture surfaces. Comparatively, the fracture surface of 718 alloy showed the typical transgranular fracture at room temperature as shown in Fig. 6b; however, at 680 °C, it showed the typical transgranular fracture in most areas and very small intergranular fracture zone located at the edge of the fracture surfaces, as shown in Fig. 7b, c.

Fig. 2 SEM micrographs of M718 alloy treated by HT1 **a**, HT3 **b**, HT4 **c**, 718 alloy treated by HT1 **d**

Fig. 3 SEM micrograph of the grain boundary precipitates **a** and corresponding EDS spectrum **b** of M718 alloy

3.3 Stress Rupture Properties

As listed in Table 4, the stress rupture lives at 680 °C and 725 MPa of M718 alloy treated by different heat treatments were all about 23 h. However, the life of 718 alloy was only 4.6 h, which was about one-fourth of M718 alloy. The stress rupture ductility of M718 alloy treated by heat

treatment HT1, HT2 and HT3 was all about 20%, which was similar to that of 718 alloy. Treated by HT4, the stress rupture ductility of M718 alloy was reduced to 11%.

The fractographs of the tested alloys ruptured at 680 °C and 725 MPa are shown in Fig. 8. The fracture surfaces of M718 alloy treated by different heat treatments showed the same typical intergranular characteristic, as shown in

Fig. 4 Morphology of Laves phase on the grain boundaries in M718 alloy **a** and corresponding SAED pattern **b**

Fig. 5 TEM images of γ'' phase and γ' phase of M718 alloy treated by HT1 **a**, HT3 **b**, 718 alloy treated by HT1 **c**, **d** SAED pattern taken from the [001] matrix zone axis of M718 alloy

Fig. 8a. And the plastic deformation was observed clearly on the exposed grain boundaries. However, as shown in Fig. 8b, c, the fracture surface of 718 alloy was composed of the transgranular zone and intergranular zone. A large number of dimples were observed clearly in the transgranular zone, and some MC carbides were located at the bottom of dimples. Nevertheless, the area of the intergranular zone was very small, and the intergranular zone was located at the outside part of the fracture surface.

Figure 9 shows the microstructures at the longitudinal section of the samples ruptured at 680 °C and 725 MPa. As presented in Fig. 9a, b, the cracks were observed to be initiated at the surfaces of all specimens and propagated along the grain boundaries toward inside. The microvoids were formed at the grain boundaries located at the inner part of both M718 alloy and 718 alloy samples, as shown in Fig. 9c, d. Comparatively, the amount of microvoids in M718 alloy was less than that

Table 3 Tensile properties of the tested alloys at room temperature and 680 °C

Alloy	Heat No.	Room temperature				680 °C			
		UTS (MPa)	YS (MPa)	EL (%)	RA (%)	UTS (MPa)	YS (MPa)	EL (%)	RA (%)
M718 alloy	HT1	1342	1005	17.0	15.9	1102	836	28.0	24.0
	HT2	1378	1081	19.3	19.1	1149	890	18.3	21.2
	HT3	1413	1121	20.7	30.1	1178	922	25.3	16.6
	HT4	1414	1120	26.0	35.0	1130	895	14.0	23.0
718 alloy	HT1	1479	1201	25.3	43.4	1121	975	19.4	16.1

Fig. 6 Fractographs of the tensile ruptured specimens at room temperature: **a** M718 alloy treated by HT3, **b** 718 alloy treated by HT1 **Fig. 7** Fractographs of the tensile ruptured specimens at 680 °C of M718 alloy treated by HT3 **a**, 718 alloy treated by HT1 **b**, **c****Table 4** Stress rupture properties of the tested alloys at 680 °C and 725 MPa

Alloy	HT No.	Life (h)	Elongation (%)
M718 alloy	HT1	22.6	16.8
	HT2	22.8	22.3
	HT3	22.6	21.0
	HT4	23.1	11.4
718 alloy	HT1	4.6	21.6

of 718 alloy, while the size of the microvoids in M718 alloy was larger than that of 718 alloy.

4 Discussion

For M718 alloy, Laves particles precipitated during the solution treatment at temperature ranging from 960 to 1050 °C in the present study. And the number of the intergranular precipitation of Laves phase decreased with increasing solution temperature. However, the solution temperature made no difference on the grain size of M718 alloy. It was implied that Laves phase precipitated during the heating up stage of the solution treatment process and hence inhibited the grain growth. The γ'' phase and γ' phase precipitated during the two-stage aging in temperature ranges of (720–750 °C) and (620–650 °C), and the sizes of

Fig. 8 Fractographs of the tested alloys stress ruptured at 680 °C and 725 MPa: **a** M718 alloy treated by HT2, **b, c** 718 alloy treated by HT1

Fig. 9 Microstructures of cross section of the tested alloys ruptured at 680 °C and 725 MPa: **a, c** M718 alloy treated by HT1, **b, d** 718 alloy treated by HT1

γ'' phase and γ' phase in M718 alloy noticeably increased with rising the aging temperatures as shown in Fig. 5.

The mechanical properties of M718 alloy were directly influenced by the microstructure or heat treatments. For HT1 and HT2, at the same solution temperature of 960 °C, increasing the aging temperatures from 720 to 750 and 620 to 650 °C, there was no difference on the intergranular

precipitation behavior of Laves phase, while the sizes of γ'' phase and γ' phase increased. As a result, the yield and ultimate tensile strengths of M718 alloy treated by HT2 were higher than those of M718 alloy treated by HT1. This seems to be consistent with the results [38, 39] that the increment of the size of strengthening phases increases the strength of the alloy since the coherency strain was the

main strengthening mechanism when the volume fraction of strengthening phases was lower than 20%. However, this could only explain that the yield strength of M718 alloy increased with increasing sizes of γ'' phase and γ' phase, but fail to explain the variation of ultimate strength with the heat treatments. Because M718 alloy treated by different heat treatments all exhibited the typical intergranular fracture, which means that the ultimate tensile strength was determined by the grain boundary strength. Apparently, the intergranular precipitation of Laves phase in M718 alloy treated by HT3 is optimum, which gave the alloy the highest ultimate strength. Although the higher solution temperature reduced the precipitation of Laves phase, too small amount of Laves phase might largely reduce the grain boundary strength. As a result, compared with that of HT3, both the ultimate strength and yield strength at 680 °C were reduced in the case of HT4 with the solution temperature of 1050 °C.

718 alloy is hardened mainly by the precipitation of γ'' phase [1–3]. A higher Nb content can improve the strength of 718 alloy remarkably [40]. The content of Nb in M718 alloy was obviously lower than 718 alloy, so more γ'' phases were expected to precipitate in 718 alloy. The grain size of M718 alloy was much bigger than that of 718 alloy. According to the Hall–Petch relationship, the strength of the alloy increases as the grain size decreases when the grain size is in the range of 0.3–400 μm [41]. Due to the above reasons, the tensile strength of M718 alloy was a little lower than that of 718 alloy.

Although both Laves phase and δ phase can refine the grain size of the alloy, the extra high addition of Mo would inhibit the recrystallization and hence increased the grain size of M718 alloy. As a result, the grain size of M718 alloy was larger than that of 718 alloy under the same rolling condition.

The stress rupture lives of M718 alloy were hardly influenced by heat treatments performed in this study. Under all of heat treatment conditions, the fracture surfaces of M718 alloy exhibited the typical intergranular fracture, so the rupture lives were determined by the initiation and propagation of the cracks at grain boundaries. It was observed that the cracks were all initiated at and propagated along the interface between Laves phase and the matrix. Thus, the lives of M718 alloy treated by different heat treatments were not different from each other.

The fracture surface of 718 alloy specimen ruptured at 680 °C and 725 MPa also exhibited the typical intergranular fracture. The microvoids formed in 718 alloy were much more than those in M718 alloy. The large amount of microvoids would largely reduce the effective area of the cross section, and finally the specimen was pulled to rupture, leaving the transgranular fracture area. Apparently,

the initiation of the cracks was very fast, so the life of 718 alloy was only 4.6 h.

The strengths of 718 alloy at room temperature and 680 °C were higher than those of M718 alloy, and the fracture surfaces of the specimens of 718 alloy exhibited the typical transgranular fracture, so the grain boundary cohesion was not the main reason for the shorter life. Compared with M718 alloy, the Mo content is very lower in 718 alloy. It is reasonable to assume that the initiation and propagation of the cracks might be influenced mainly by the diffusion rate of the elements. The addition of Mo as high as 7.50 wt% largely reduced the diffusion rate when the alloy stressed at 680 °C and 725 MPa, and hence prolonged the rupture life of the alloy noticeably. In addition, the finer grain size also favored the initiation of the intergranular cracks during the stress rupture test, and hence, the conclusion could not be drawn directly that the life of M718 alloy was much longer than that of 718 alloy. However, the life was only 45 h for 718 alloy ruptured at 650 °C and 690 MPa, and 53 h for 718 alloy ruptured at 680 °C and 540 MPa [42]. It is reasonable to believe that the addition of Mo has a noticeable effect on the stress rupture life of 718 alloy.

The fracture surfaces of the tensile and stress rupture specimens of M718 alloy showed the intergranular fracture characteristic in this study. It indicated that the grain boundary strength of M718 alloy was always lower than the intragranular strength. However, the fracture surfaces of the specimens of 718 alloy were transgranular fracture at room temperature and mixed fracture at high temperature, which were consistent with the previous study [43]. It was shown that the grain boundary strength of 718 alloy was higher than the intragranular strength at room temperature. As the temperature increased, the grain boundary strength decreased quickly. When the grain boundary strength was lower than the intragranular strength, the fracture surface of 718 alloy specimen was mixed mode.

5 Conclusions

1. Laves phase precipitates at the grain boundaries of M718 alloy instead of δ -Ni₃Nb phase in 718 alloy, and γ'' phase and γ' phase precipitate in the matrix of M718 alloy as that of 718 alloy.
2. Increasing the solution temperature from 960 to 1050 °C noticeably reduces the intergranular precipitation of Laves phase, and rising the two-stage aging temperatures markedly increases the sizes of γ'' phase and γ' phase.
3. The strength of M718 alloy increases by increasement of the sizes of γ'' phase and γ' phase and appropriate precipitation of Laves phase at the grain boundaries.

The stress rupture life of M718 alloy is not influenced by heat treatments, because the cracks are initiated and propagated at the Laves/matrix interface along the grain boundaries.

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