Influences of γ'' and δ Precipitations on the Microstructural Properties of 718 Alloy through HIP, Solid-Solution, and Different Aging Heat Treatments

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In our previous work, the parameters of solid-solution treatment for 718 alloy were discussed. The results showed that a solution temperature of 1020°C is optimum. The Laves and δ phase can complete solution after 1020°C, 1 h solid-solution treatment. The aim of this study is to discuss the effect of various aging treatments for as-HIP treated 718 alloy. In the experiment, seven different aging treatments were used: 720°C for 2, 4, 8 and 16 h, furnace cooled to 620°C, soak for 8 h, and air cooled to room temperature; 720°C for 8 h, furnace cooled to 620°C, soak for 2, 4, 8 and 16 h, and air cooled to room temperature. Experimental results showed that 1020°C solid-solution, 720°C for 8 h and 620°C for 4 h of aging treatment for as-HIP treated 718 alloy is optimum. All the precipitations are MC carbides; the dimension of γ'' precipitations is 30.41 nm, which with a maximum tensile strength (1407.2 MPa) and elongation (14.5%) at room temperature.
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1. Introduction

Nickel base alloy castings generally are made by the investment casting process. 718 alloy is a nickel base superalloy commonly used in the fabrication of critical pieces for turbine engines, because of its high mechanical properties, and good corrosion resistance.^{1,2)} Precipitation-strengthening nickel alloys contain aluminum, titanium, or niobium to cause precipitation of a second phase during appropriate heat treatment. The precipitated phase, usually γ' or γ'' , substantially increases the strength and hardness of the alloy. However, 718 alloy strengthened by the γ'' intermetallic precipitate (Ni_3Nb), has become and will continue to be the workhorse alloy for aircraft engines.^{3–6)}

HIP processes take place under high pressure and high temperature conditions. It applies heat and pressure within an enclosed vessel to consolidate or dense materials such as castings. The technology is a common method in the industry to improve strength and durability of the parts and remove voids from castings.^{7–9)} Its major application is for eliminating casting defects and improving mechanical properties. By utilizing this advantage, rapid development in the use of HIP in cast superalloy was well known.^{4,5,10–12)}

The temperature and time parameters of heat treatment would significantly affect the strengthening phase of γ'' precipitation, or the harmful phase such as Laves and δ phases for the precipitation-strengthening 718 alloy. Therefore, this study aimed to discuss the feasibility of solid solution and aging heat treatments, especially on the effects of different temperatures and soaking times of aging treatment on the precipitates, as well as the advantages and disadvantages of mechanical properties.

In earlier studies, Chang *et al.* reported the effects of different parameters of HIP treatments for 718 alloy.^{3,4,10,11)} The results showed that the HIP treatment for 718 alloy at

1180°C, 175 MPa, and 4 h is the optimal process. They also pointed out that the optimal solid-solution temperature for 718 alloy is 1020°C.¹³⁾ As a result, the hardness can increase to $H_{V0.3}$ 506.7, tensile strength to 1331.5 MPa, and elongation to 6.1% after optimal HIP, 1020°C solid-solution and 720°C 8 h, 620°C 8 h aging treatments. However, the solid solution and aging treatments would obviously affect the precipitation and properties of 718 alloy. This study further examined the effects of the microstructure and mechanical properties of 1020°C solid solution and different aging temperature treatments for as-HIP treated 718 alloys.

2. Experimental Procedures

In this study, the chemical composition (mass%) of 718 alloy is as follows: 52.89% Ni, 19.36% Fe, 17.30% Cr, 5.26% Nb, 3.52% Mo, 0.83% Ti and 0.52% Al. 718 alloys were prepared by VIM process. After melting, the alloys were solidified into square plate castings. The microstructure observed and tension test of specimens obtain from the castings by wire electrical discharge machining. 718 alloys were chosen to carry out the HIP and heat treatments. HIP equipment was from USA Flow Pressure Systems,¹⁴⁾ after HIP treatment, solid solution and age hardening treatments were performed in an atmosphere furnace.

In this experiments, the solid-solution temperature was fixed at 1020°C, under eight different aging processes: 720°C for 2, 4, 8, and 16 h, furnace cooled (55°C h^{-1}) to 620°C, then soaked for 8 h, and air cooled to room temperature are designed as “A2-8”, “A4-8”, “A8-8” and “A16-8” respectively; 720°C for 8 h, furnace cooled to 620°C, soaked for 2, 4, 8, and 16 h, and then air cooled to room temperature are designated as “A8-2”, “A8-4”, “A8-8” and “A8-16” hereafter.²⁾ To evaluate the effects of aging treatments on 718 alloy, microstructure inspections, hardness and tensile tests were performed. In addition, EDX, SEM and TEM inspections were carried out.

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Microhardness tests were measured by HV with loading up to 300 g and in compliance with the CNS 2115 Z8004 standard. The specimen sizes of the tension test are follow-up the standard of CNS 2112 G2014. For the tension test, a SHIMADZU universal material test machine with a maximum load of 25 tons was used and strain rate was 0.001 s^{-1} at room temperature.

3. Results and Discussion

3.1 Examination of aging treatments on the microhardness

Based on the previous study, the optimal solid-solution temperature is 1020°C , 1 h for as-HIP treated 718 alloy. In order to investigate the effects of a two-step aging treatment, the following section will discuss two aging treatments. The first step of aging temperature is 720°C , and second step of aging temperature is 620°C . All specimens in this study were performed by the HIP process and 1020°C solid-solution heat treatment.

The main purpose of the solution is a complete solid solution of niobium for γ' and γ'' precipitations. Meanwhile, harmful phases, such as Laves and δ , can be re-dissolved within the γ matrix. In general, heat treatment process allows the solid solution of precipitations to acquire a homogeneous microstructure, and then strengthen the elements by growing uniform precipitation during aging treatment. In the first step of the aging treatment, there is more nucleation energy to form each precipitation at the higher temperature. However, in order to avoid the coarsening phenomenon of the strengthening phase, the second step of the aging treatment should be used in lower temperature, which is suitable for the growth of precipitations.^{1,2)} Then the precipitations can maintain the homogeneous distribution of grain size. As a result, the mechanical properties of the 718 alloy can be improved.

Figure 1 shows the microhardness test results of A2-8, A4-8, A8-8 and A16-8 specimens. The average microhardness data were obtained at least six areas for each specimen and three test specimens for each experimental condition. Increasing the soaking time in the first step of the 720°C aging treatment ($2 \rightarrow 4$ h) would increase the hardness. The maximum hardness was $H_{V0.3}$ 547.68, which appeared in the A4-8 specimen. However, by continuing to increase the soaking time for the first step of 720°C aging treatment ($8 \rightarrow 16$ h), the microhardness showed a downward trend. When the soaking time was over 8 h at 720°C aging, some of the δ precipitations appeared in the γ matrix, which were detrimental to the strengthening phase, and resulted in a lower strength as shown in Fig. 2.¹⁵⁾ Thus, the hardness of A16-8 specimen is the lowest, and decreased to $H_{V0.3}$ 481.79.

As the main precipitated strengthening phase of 718 alloy is γ'' , by increasing the amount of γ'' , the precipitations would result in higher hardness.^{11,13)} Moreover, the nucleation energy of γ'' would be enhanced by an increased soaking time in the first step of the aging treatment, and then, grew to a more appropriate size in the second step. Figure 1 also shows that the microhardness of the A4-8 specimens was higher than the A2-8 specimens. Our early study^{8,10,11)} pointed out that the grain would grow and coarsen at higher

Fig. 1 Comparison of microhardness of the A2-8, A4-8, A8-8 and A16-8 specimens.

Fig. 2 SEM micrographs of the A8-8 specimens.

temperatures; larger γ'' showed a growing trend. On the contrary, compared to smaller γ'' , the precipitation would be dissolved. The nucleation and growth of γ'' phase is a dynamic process, which means that the processes of aging treatments are to increase nucleate points and γ'' growth as simultaneous formations. With continued increase of the soaking time for the first step of aging treatment (the A8-8 specimens), even though there would be more γ'' phase precipitation, part of the γ'' phase is excessive growth, and it would still result in lower hardness as shown in Fig. 1.

Figure 3 shows the comparison of microhardness of the A8-2, A8-4, A8-8 and A8-16 specimens. Obviously, increasing the soaking time of the second step of aging treatments was insignificant help for strengthening of the 718 alloy. However, only a little variation in microhardness could also affect the tensile properties, the maximum hardness was $H_{V0.3}$ 517.61, which appeared in the A8-4 specimens. Increase the soaking time to 16 h for the second step of aging treatments would still result in the lowest hardness ($H_{V0.3}$ 424.18). According to the AMS5596 standard,¹⁶⁾ the minimum requirement of hardness for 718 alloy is HB 331 (H_V 350). All hardness degrees of specimens are up to AMS5596 standards. However, as described previously, the longer soaking times of aging treatments produces no better results. The following discussions will focus only on 2 and 4 h for the first and second steps of aging treatments.

Fig. 3 Comparison of microhardness of the A8-2, A8-4, A8-8 and A8-16 specimens.

Table 1 The value of f_i for different elements.

Element	Fe	Cr	Al	Ti	Mo	Nb
f_i	0.0119	0.013	0.0183	0.036	0.0421	0.0624

Table 2 Composition of the γ phase for different aging treatments (at%).

Aging treatment	Al	Nb	Mo	Ti	Cr	Fe	Ni
A2-8	1.44	2.33	1.86	1.63	18.68	19.18	Bal.
A4-8	1.42	1.82	1.67	1.08	18.53	18.02	Bal.
A8-2	1.48	2.68	2.29	1.92	15.74	21.65	Bal.
A8-4	1.16	3.14	1.61	1.79	16.26	21.77	Bal.

Fig. 4 SEM micrographs of (a) A2-8, (b) A4-8, (c) A8-2, (d) A8-4.

3.2 Examination of aging treatments on the microstructure characteristics

Figure 4 shows the SEM micrographs of the A2-8, A4-8, A8-2 and A8-4 specimens. The result confirms that Laves and δ phases were complete solid solution at 1020°C. They did not precipitate in the matrix after a short time of aging treatment. The microstructure was only few changed after 2 or 4 h of soaking time. According to the EDX analysis, all the precipitations are MC carbides. M. G. Burke¹⁷⁾ reported that the composition of γ would be different because the amount of precipitation is changed for the 718 alloy. In addition, W. C. Liu *et al.*¹⁸⁾ calculated the lattice constant by using the composition of γ matrix, as shown in eq. (1), where a_{Ni}

is the lattice constant of Ni as (0.35234 nm), and f_i is the coefficient of the alloy element (as shown in Table 1), and D_i is the atomic percentage of the element.

$$a = a_{Ni} + \sum f_i D_i \quad (1)$$

The compositions of the γ phase of different aging treatments are shown in Table 2. Moreover, the calculated results for lattice constant are shown in Table 3. Experimental results showed that the lattice constant would increase with an extended soaking time in the first and second steps of aging treatments. Previous study indicated¹³⁾ that increasing the solution temperature would enhance the solid-solution volume of niobium, and cause the lattice to be deformed.

Table 3 The lattice constant of different aging treatments (nm).

Aging treatment	Lattice constant (nm)
A2-8	0.36023316
A4-8	0.36035253
A8-2	0.36038732
A8-4	0.36043668
A8-8	0.36050667

Table 4 The weight percentage of the γ'' phase for different aging treatments (mass%).

Aging treatment	γ'' (mass%)
A2-8	3.08
A4-8	3.36
A8-2	8.57
A8-4	10.6
A8-8	12.42

Fig. 5 TEM micrographs of γ'' precipitation observed on (a) A2-8, (b) A4-8, (c) A8-2, (d) A8-4.

Likewise, increasing the soaking time of an aging treatment would enlarge the lattice constant. In this study, the minimum lattice constant (0.36023316 nm) appeared in the A2-8 specimens.

W. C. Liu¹⁸⁾ used the lattice constant of γ phase, and calculated the weight percentage of γ'' and γ' , as shown in eq. (2).

$$a = a_0 - \frac{n_1 p_1 W_{\gamma''} + n_2 p_2 W_{\gamma'}}{m(m - n_1 p_1 W_{\gamma''} - n_2 p_2 W_{\gamma'})} \quad (2)$$

Furthermore, R. B. Li *et al.*^{19,20)} also reported that the γ' precipitations amounted to only one-third of the γ'' precipitations. Table 4 shows the calculated results of the weight

percentage for the γ'' phase under different aging treatments. Due to the fixed first step, the soaking time would provide the similar nucleation and size of γ'' precipitations. Moreover, a lower temperature in the second step of soaking time (620°C) would cause a slow growth in the size of the γ'' phase. Therefore, by increasing the soaking time of the second step, the $W_{\gamma''}$ are enhanced, which could significantly affect the tensile strength.

Figure 5 shows the TEM micrographs of γ'' precipitation observed of the A2-8, A4-8, A8-2 and A8-4 specimens. The narrow black area represents the γ'' precipitations. The γ'' nucleates heterogeneously at the γ'/γ interface, and tends to merge with γ' particles, thus creating a γ'/γ'' co-precip-

Fig. 6 Comparison of the dimensions of γ'' precipitations on 718 alloy through HIP, 1020°C solid-solution, and different soaking times of aging treatments.

itate.¹⁰⁾ In fact, the γ'' phase is the predominant strengthening precipitation of 718 alloys. Merrick pointed out²¹⁾ that can use the TEM micrographs to measure and calculate the size of γ'' precipitations. In this study, we also used the TEM micrographs and according this report to calculate the average dimension of γ'' precipitations. The calculated average dimensions of γ'' precipitations are shown in Fig. 6, where the short soaking time (A2-8 specimen) would possess the smallest dimensions of γ'' precipitations (16.82 nm). Figure 6 shows the dimensions of γ'' precipitations, which are increased with the extended soaking time, especially under higher temperature conditions (in the second step). Literature²²⁾ pointed out that, at different aging treatments, the size of the γ'' phase has a linear relationship with the cube root of soaking time. Therefore, the dimensions of γ'' precipitations were increased (30.41 nm) after the 720°C 8 h, 620°C 4 h aging treatment (A8-4), which matches the results of extended soaking times resulting in increasing the weight percent of γ'' precipitations as listed in Table 4.

3.3 Effect of aging treatments on the tensile properties

Figure 7 shows the mechanical properties of the A2-8, A4-8, A8-2, A8-4 and A8-8 specimens. The results as compared with the BM (base metal), and showed that all aged specimens have higher strength and ductility except for the A8-8. The γ'' precipitations are helpful to improve the

hardness and strength for the 718 alloy. However, the coarsen γ'' precipitations would result in lowered strength after extended soaking time as shown in Figs. 1 and 3. Jianhong *et al.*²³⁾ pointed out that the aging treatment would lead the γ'' to the coarsening phenomenon. Meanwhile, 718 alloy would achieve the maximum strength under a γ'' phase up to a certain size. Extending the soaking time for the second step of an aging treatment (A8-8) would increase the weight percentage of the γ'' phase (Table 4), and reasonably result in a coarse precipitation. Figure 7(a) can further compared with the data as shown in Fig. 6. The tensile strength tends to increase as the dimension of the γ'' precipitations increased. However, Fig. 7(b) shows the elongation is obviously decrease in the A8-8 specimens. This results show that the other microstructure factors, except for γ'' precipitations will obviously affect the elongation. To examine the other microstructure factors, Fig. 2 shows the δ precipitations significantly affect the tensile properties, which resulted in decreasing elongation. Figure 7 also shows the coarsening γ'' precipitations are a slight effect on tensile strength. It is possibly to argue that the contribution of the elongation in aged specimens may combine an increase particle size of γ'' precipitations with the appearance in δ precipitations. As a result, the elongation increases slightly and then significantly decrease after the soaking time for the second step of an aging treatment performed. The better elongation (14.5%) appeared in the A8-4 specimens. In addition, the optimal yield strength and tensile strength were 1280.44 MPa and 1407.22 MPa respectively. It is reasonable to suggest that the heat treatment in the A8-4 specimens was the peak aging treatment. Consequently, the optimal parameters of heat treatment for 718 alloy was 1020°C 1 h solid-solution treatment, 720°C for 8 h, and furnace cooled to 620°C for 4 h, then air cooled to room temperature.

4. Conclusions

- (1) The optimal aging treatment for as-HIP treated 718 alloy is 720°C 8 h, 620°C 4 h. Meanwhile, the maximum tensile strength is 1407.22 MPa, yield strength is 1280.44 MPa, and elongation is 14.5%, respectively.
- (2) The Laves and δ phases are complete solid solution at 1020°C, and are not precipitated in the matrix after the

Fig. 7 Comparison of the mechanical properties of the A2-8, A4-8, A8-2, A8-4 and A8-8 specimens (a) tensile strength, (b) elongation.

aging treatment. According to the EDX analysis, the precipitations are MC carbides after 1020°C solid-solution and aging treatments.

- (3) Extending the soaking time for the second step of the aging treatment would increase the weight percentage of the γ'' phase, but result in a coarse precipitation and lower hardness. Consequently, the coarse γ'' precipitations affect the tensile strength, and a few δ precipitations appeared in the matrix also result in decreasing the elongation.

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