QUANTITATIVE CHARACTERIZATION OF TWO-STAGE HOMOGENIZATION TREATMENT OF ALLOY 718

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Abstract

Segregation behavior of a ϕ406 mm Alloy 718 ingot was investigated and two-stage homogenization treatment was employed to relieve the micro-segregation between dendrite structures. In the first stage homogenization, the Laves phase elimination percentage was expressed dependence of two parameters (time and temperature). In order to characterize the effects of second stage homogenization, a 900 /1 h treatment was conducted after homogenization, which could form δ-phase (Nb-riched) to trace the diffusion of Nb. It was found that average composition of δ-phase area, quantity of δ-phase precipitation and size of δ-phase precipitation would change with the proceeding of homogenization. As a result, homogenous structure can be achieved by means of a two-stage homogenization treatment (1140 /60 h + 1190 /30 h).

Introduction

Alloy 718 has a wide application in aircrafts and land-based gas turbine engines because of the outstanding fabrication performance, excellent welding properties and reasonable cost [1-3]. One of the chemical characteristics for Alloy 718 is about 5.5 mass% Nb addition, which forms tetragonal DO22-structure γ″ (Ni_3Nb) to increase the strength at elevated temperatures (~650) [4-5]. However, such high concentration of Nb will undoubtedly cause severe segregation during solidification and increase the difficulty in the subsequent homogenization treatment.

Recently, more and more studies put emphasis on two-stage homogenization treatment for wrought superalloys. In the first stage, hiring temperature is a little lower to avoid the melting of inter-dendritic precipitates. Specifically, Laves phase in Alloy 718 should be eliminated after the first stage homogenization. Hence, development of Laves phase elimination models would have both scientific and technical significance, which will provide an effective and economical tool for homogenization designs. The previous works have suggested that alloying elements still segregate between the dendrites even after the first stage homogenization treatment [6-7]. So, second stage homogenization at higher temperature is necessary to accelerate the diffusion of alloying elements. However, it is difficult to determine the locations of dendrite core and inter-dendritic region upon the previous experimental results, which puts big obstacle to verify the elements diffusion. Here, δ-phase (Nb-enriched) is selected to trace the diffusion of Nb, which is also named as "Tag" method. In X. Liang *et al*'s research [8], "Tag" method is applied to investigate the segregation status of Alloy 718 after different temperature homogenization

(1180 ℃/63 h, 1120 ℃/52 h and etc). But no detailed work is concerned with homogenization at one certain temperature continuously.

In view of the research status mentioned above, we try to characterize the effects of two-stage homogenization of Alloy 718 quantitatively in this study, which will give some insights on homogenization designs of Alloy 718.

Experimental Methods and Materials

φ406 mm Alloy 718 ingot with overall compositions listed in Table Ⅰ was produced by doubled melting technique (VIM+VAR). Fig.1a shows the investigated ingot and typical dendrite structure of as-cast alloy is shown in Fig.1b.

Table Ⅰ Chemical compositions of Alloy 718 superalloy (mass %)

C	Si	Al	Ti	Mo	Nb	Cr	Ni	Fe
0.05	0.07	0.50	1.00	3.30	5.35	19.5	53.0	Bal.
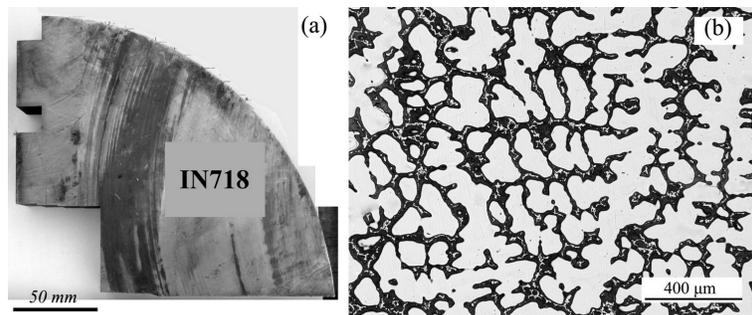
Figure 1. Optical micrographs showing:
(a) 718 superalloy used in this study, (b) As-cast dendrite structure.

The samples (20×5×5 mm) were cut from the center of ingot for homogenization. The parameter of homogenization was generated according to the incipient melting temperature determined by metallographic method. Then, two-stage homogenization was conducted (1140 ℃/60 h + 1190 ℃/30 h). As for Alloy 718, 900 ℃ is the peak of δ-phase precipitation [9-10]. And δ-phase is a kind of Nb-enriched phase, which can be utilized to depict the changes of Nb segregation. In this study, a 900 ℃/1h treatment was conducted subsequently on five samples listed in Table Ⅱ to characterize the effects of the second stage homogenization.

Table Ⅱ Parameters of homogenization treatment prior to 900 ℃/1 h.

Samples	Homogenization treatment criterion
1#	1140 ℃/50 h
2#	1140 ℃/60 h
3#	1140 ℃/60 h + 1190 ℃/10 h
4#	1140 ℃/60 h + 1190 ℃/20 h
5#	1140 ℃/60 h + 1190 ℃/30 h

The optical microscope samples were prepared by mechanical polishing and electro-etching in a solution of 11 vol.% hydrochloric acid, 33 vol.% nitric acid and 56 vol.% glycerin at a voltage of 6 V for 8 s. Afterwards, the optical microscopy and scanning electron microscopy-energy dispersive spectrometry (SEM-EDS) examinations were carried out to characterize the microstructure of the samples. Here, metallographic analysis software was used to calculate the variation of Laves phase area percentage during homogenization. The differential scanning calorimeter (DSC) examination was performed to verify the elimination of Laves phase and the heating rate was controlled at 10 /min from 1,100   to 1,400 .

Results and Analysis

Incipient Melting Temperature (IMT)

Metallographic method is applied to determine the IMT in this study. It is reported that the IMT of ɸ423 mm Alloy 718 ingot is around 1,163 ~1,175   [8]. In reference with this result, the samples are soaked at 1,150 , 1,160 , 1,170 , 1,180   and 1,190   for 30 minutes and then quenched in water. Fig.2 shows the microstructure and Laves phase morphology evolution during heating process. From Fig.2a-c, no obvious change has been found concerning to the appearance of Laves phase with the increase of soaking temperatures. When the soaking temperature is raised to 1,180 , incipient melting appears in some areas of Laves phase. When 1,190   is reached, γ/Laves eutectic is observed. This phenomenon indicates that all of the Laves phase has been turned into liquid when soaked at 1,190   and the appearance of γ/Laves eutectic is the result of water quenching. From the above analysis, it can be inferred that the IMT of ɸ406 mm Alloy 718 ingot is ranged approximately from 1,170   to 1,180   and then the subsequent homogenizing temperature should be lower than 1,170 .

Quantitative Analysis of Laves Phase Elimination

Previously, it has been revealed that the homogenizing temperature should be lower than 1,170 . Here, 1,140   is chosen as the experimental temperature and there are two main reasons to be addressed. First, real heating temperature in industry will be fluctuated around the setting temperature because of the heating furnace itself and interference from outside environment. So, it is greatly important to investigate homogenization process at a bit lower temperature in order to have an overall command of homogenization technique, which will be valuable to industry producing. Second, homogenization data at 1,140   can be utilized to establish model in present study for higher temperatures prediction. Based on the two reasons above, 60 h homogenization at 1,140   is conducted and quantitative analysis of the Laves phase is applied to evaluate the homogenization effects.

Fig.3 shows the microstructure evolution from 3 h to 40 h. With the proceeding of homogenization, both dendrite structure and Laves phase disappears. When the homogenizing time reaches 30 h, little Laves phase still exists while no Laves phase except for MC carbide is observed after 40 h, shown in Fig.3d-e.
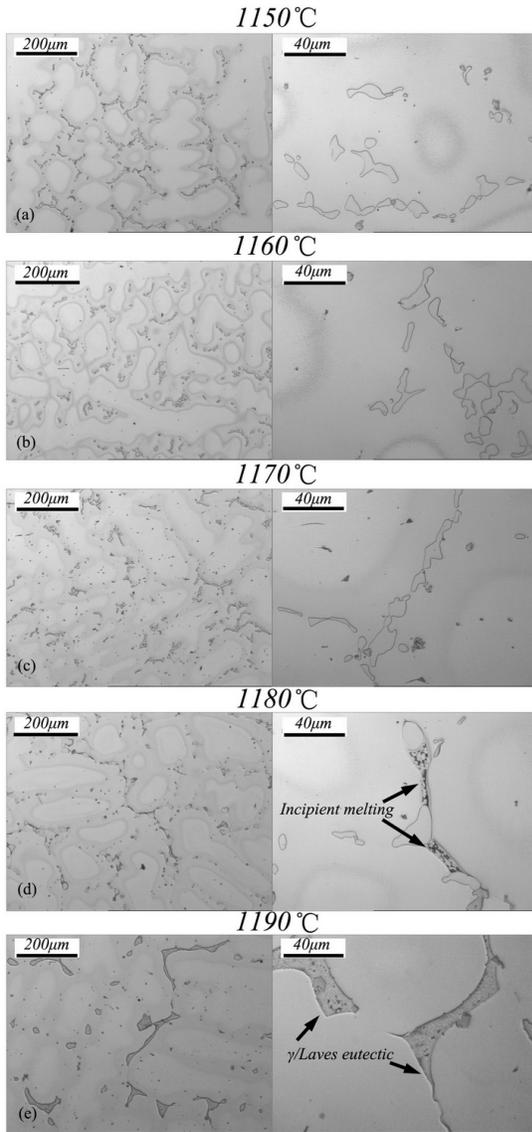
Figure 2. Microstructure of as-cast alloy after soaking at (a) 1,150 , (b) 1,160 , (c) 1,170 , (d) 1,180 and (e) 1,190 for 30 minutes then quenched in water.

Figure 3. Microstructure evolution during the homogenization at 1,140℃. (a) 3 h, (b) 5 h, (c)10 h, (d) 30h, (e) 40h.

Furthermore, it can be inferred that Laves phase disappears between 30 h and 40 h. To be estimated, we use 35 h to define the time to eliminate Laves phase completely. After quantitative metallographic analysis, the Laves phase area percentage of as-cast alloy is 3.2% and variation of that during homogenization is illustrated in Fig.4. Apparently, this curve displays an exponential function, where used Eq.(1) for fitting. When $t=0$, the value of $C_{(Laves)}$ is 0.032, so A=0.032.

$$C_{(Laves)} = A\exp(-K_T t) \tag{1}$$

$$C_{(Laves)} = 0.032\exp(-K_T t) \tag{2}$$

$$C_{(Laves)}^{1140°C} = 0.032\exp(-0.222t) \tag{3}$$

Where t (hour) is the homogenizing time. Eq.(3) presents the relationship between Laves phase area percentage and homogenizing time at 1,140 .

In the previous research, Eq.(4) is used for predicting the time to eliminate Laves phase completely in φ423 mm Alloy 718 ingot [8,11], which can be applied in this case.

$$\tau = B\exp(-0.036T) \tag{4}$$

Where τ (hour) is the time to eliminate Laves phase completely; T ( ) is the homogenizing temperature; the coefficient B is decided by the segregation degree of alloy ingots. In this study, the time to eliminate Laves phase at 1,140 for 406 mm Alloy 718 ingot is 35 h, so B=2.33×10^{19}
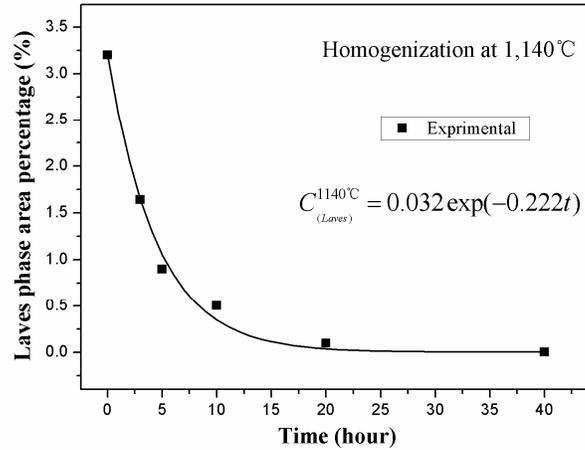
Figure 4. Laves phase area percentage as a function of time during homogenization at 1,140 .

Based on the Eq.(1), we can rationally assume that the value of K_T is only related with homogenizing temperature. That means if the temperature is determined, K_T is fixed. In order to predict the Laves phase elimination process at higher temperatures, Eq.(2) and Eq.(4) are used to deduce the following equations:

Eq.(2) can be written as:

$$K_T = \frac{\ln 0.032 - \ln C_{(Laves)}}{t} \tag{5}$$

It is assumed that the process of Laves phase elimination is over when the Laves phase area percentage is 0.01%. Then

$$t = \tau = A\exp(-0.036T) \tag{6}$$

Put Eq.(6) into Eq.(5), we can get:

$$K_T = \frac{\ln 0.032 - \ln 0.0001}{B\exp(-0.036T)} = \frac{5.768}{2.33 \times 10^{19}\exp(-0.036T)} = \frac{2.48 \times 10^{-19}}{\exp(-0.036T)} \tag{7}$$

Combined Eq.(2) and Eq.(7), an integrated equation could be derived.

$$C_{(Laves)} = 0.032\exp[(-2.48 \times 10^{-19}t)/\exp(-0.036T)] \tag{8}$$

Here, another parameter (Laves phase remaining percentage, E (%)) is defined as follows:

$$E_{(Laves)} = \frac{C_{(Laves)}}{0.032} = \exp[(-2.48 \times 10^{-19}t)/\exp(-0.036T)] \times 100\% \tag{9}$$

It can be known that the value of E (%) is 100 before homogenization (t=0) while zero after eliminating Laves phase completely.

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Based on the above Laves elimination equation, the elimination process of Laves phase could be predicted in quantity at certain temperature for certain time during homogenization.

Second Stage Homogenization Characterization

Fig.5 shows the distribution of δ-phase in different homogenization treatments after 900 /1 h. The grey parts are δ-phase precipitation area and white parts are area free of δ-phase precipitation. There are two kinds of δ-phase available as shown in Fig.6, which are rod-shaped and particle shape. Rod-shaped δ-phase is always found around MC carbide. The main composition of MC carbide is Nb, which will certainly promote the precipitation and growth of δ-phase around MC carbide. A majority of δ-phase are particle shape precipitate on the matrix shown in Fig.6b.
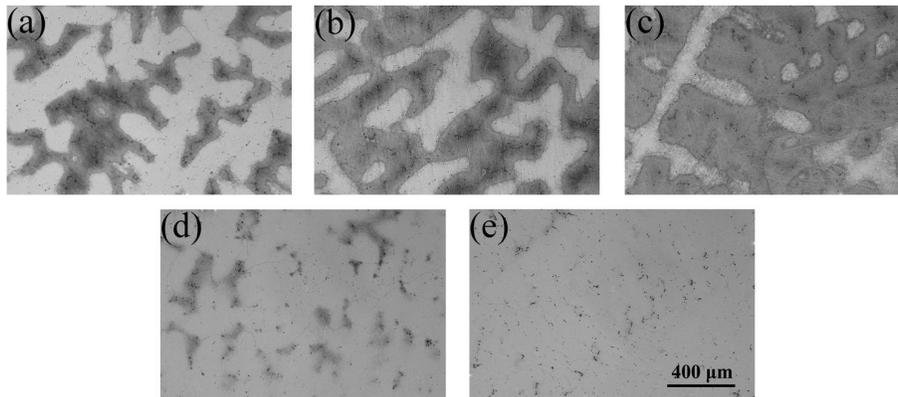
Figure 5. δ-phase distribution after different homogenization treatments and then 900 /1 h. (a) 1140 /50 h, (b) 1140 /60 h, (c) 1140 /60 h + 1190 /10 h, (d) 1140 /60 h + 1190 /20 h, (e) 1140 /60 h + 1190 /30 h.
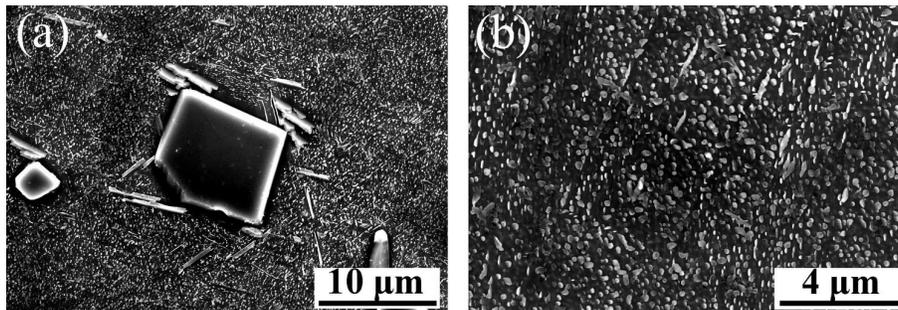
Figure 6. δ-phase precipitation in sample 2# (1140 /60 h) after 900 /1 h. (a) Rod-shaped δ phase precipitation around MC carbide, (b) Particle δ phase.

The variation of δ-phase area percentage in different homogenization treatments is illustrated in Fig.7. To be apparent, two steps can be summarized concerning the precipitation of δ-phase. Firstly, δ-phase area percentage is increasing with the proceeding of homogenization. Especially for sample 3# (1140 /60 h + 1190 /10 h), 83.2% of total area is δ-phase precipitation area. However, the increasing tendency stops when the second stage homogenization time arrives at 20 h. The number drops dramatically to 11.8% and 1.5% for sample 5# (1140 /60 h + 1190 /30 h), eventually. This interesting phenomenon would be discussed in the last section.
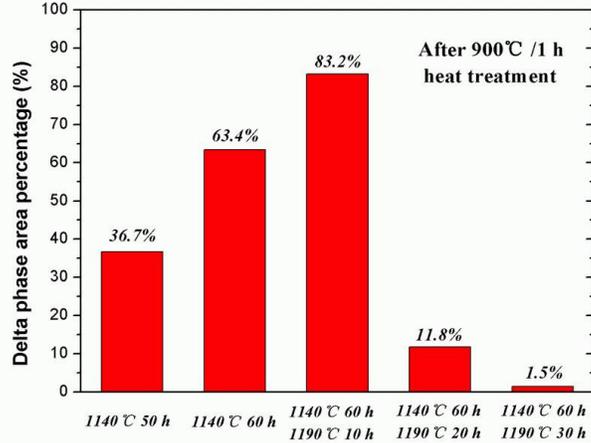
Figure 7. δ-phase area percentage in different homogenization treatments after 900 /1 h.

In addition to the investigation on δ-phase area percentage, elements distribution is also studied in terms of two distinct areas (δ-phase area and free of δ-phase area). The average compositions (mass %) of four elements are presented in Table Ⅲ. It is shown that Nb concentration in δ-phase area is 6.5 mass % while 5.0 mass % in free of δ-phase area. But, with the proceeding of homogenization, the average Nb concentration in δ-phase area drops and approaches the nominal alloy composition, finally. In contrast to the visible fluctuation of Nb concentration, Mo concentration almost has no big change. It is inferred that the Mo concentration has been homogenous after 1140 /40 h in the first stage because of its low segregation behavior compared with Nb. It is also noted that the diffusion behavior of Cr shares the same pattern with Fe.

Table Ⅲ Average compositions (mass %) in δ phase area and free of δ phase area after different homogenization treatments.

Samples	Nb		Mo		Cr		Fe	
	Area 1	Area 2	Area 1	Area 2	Area 1	Area 2	Area 1	Area 2
1#	5.14	6.44	3.28	3.36	20.30	18.83	19.10	17.56
2#	5.26	6.08	3.30	3.41	20.20	19.26	18.63	17.62
3#	5.34	5.85	3.23	3.31	19.98	19.41	18.41	17.88
4#	5.42	5.68	3.29	3.38	19.67	19.51	18.32	17.93
5#	5.45	5.58	3.30	3.33	19.55	19.56	18.10	18.01

*Area 1 is free of δ-phase area; Area 2 is the δ-phase area.

Fig.8 displays the morphology of δ-phase area in different homogenization treatments after 900 /1 h. It is important to point out that the quantity and size of δ-phase can be used to evaluate the segregation evolution apart from the composition variation. For sample 1# (1140 /50 h), large particle δ-phase as well as some rod-shaped δ-phase can be seen from Fig.8a. While for sample 3# (1140 /60 h + 1190 /10 h), the particle δ-phase is smaller in size and the density of particle is also smaller. Only a little δ-phase appear for sample 5# (1140 /60

h + 1190 /30 h),. The whole evolution of δ-phase precipitation in quantity and shape indicate that Nb concentration tends to be homogeneous after two-stage homogenization treatment.

Finally, we can look back to the interesting phenomenon stated before. It can be seen from Fig.7 that the area percentage of δ-phase reaches the highest for sample 3# (1140 /60 h + 1190 /10 h). This can be explained after understanding chemical conditions of δ-phase precipitation. Here, it is supposed that the Nb concentration is a key factor of δ-phase precipitation. That means δ-phase precipitates when the Nb concentration is higher than a certain value. If the Nb concentration is lower than this value, no δ-phase will precipitate. Similarly, if the Nb concentration is relatively higher, the δ-phase would be large in size. Following this assumption, with the diffusion of Nb, more and more areas meet the chemical requirement for the precipitation of δ-phase, which results in the rising trend of δ-phase area percentage shown in Fig.7. When the diffusion of Nb keeps going on, a majority of areas do not meet the Nb concentration requirement for the precipitation of δ-phase. Naturally, the precipitation quantities drop dramatically.
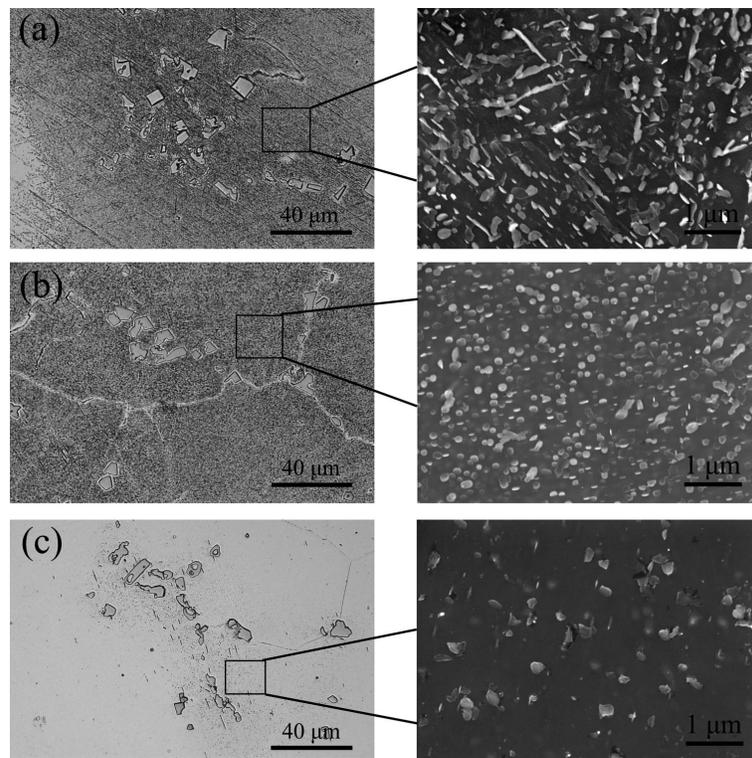
Figure 8. Morphology of δ phase area after different homogenization treatments and then 900 /1 h. (a) 1140 /50 h, (b) 1140 /60 h + 1190 /10 h, (c) 1140 /60 h + 1190 /30 h.

Conclusions

1. Incipient melting temperature of a φ406 mm Alloy 718 ingot is in the range of 1,170 ~1,180 .

2. The Laves phase remaining percentage is derived as a function of homogenizing temperature (T,) and homogenizing time (t, hour): $E_{(Laves)} = \exp[(-2.48 \times 10^{-19} t)/\exp(-0.036T)] \times 100\%$

3. Alloying elements distribution tends to be homogeneous eventually by means of two-stage homogenization treatment, judging from three aspects: average composition of δ-phase area, quantity of δ-phase precipitates, and size of δ-phase precipitates, respectively.

Acknowledgments

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