AN INVESTIGATION OF THE HOMOGENIZATION

AND DEFORMATION OF ALLOY 718 INGOTS

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ABSTRACT

The segregation of Alloy 718 ingots and the technical factors which affect the ingot homogenization practice are studied in this paper. The segregation and melting point of Alloy 718 ingots are studied by DTA, electron microprobe and structure analysis. The elimination of Laves phase is studied in detail. After the solution of Laves phase the segregation elimination of Nb is reviewed further. Through the investigation an optimum homogenization treatment to eliminate the segregation of Alloy 718 ingots is proposed. Furthermore, the affection of deformation on homogenization practice of Alloy 718 ingots has been also studied.

Introduction

Since Alloy 718 was developed by the International Nickel Company in the 1950's the problem of segregation has existed in the productions and applications of Alloy 718. The content of element Nb in Alloy 718 is over 5 wt. %, it will form a serious segregation during the solidification of the alloy ingots. Although the melting process is more and more advanced and the many methods have been used to alleviate the segregation, for example, Institute of Metal Research, ShenYang, China, adopts strickly controlling the contents of minor elements P, B, Zr and Si in superalloy, to alleviating the solidification segregation with great success, the segregation in Alloy 718 ingots is still serious. To eliminate the segregation, the homogenization practice must be used, and current applications for Alloy 718 are placing emphasis on improving the alloy's properties and quality by thermomechanical processing (TMP). The key to successful TMP is free of segregations in the alloy. Therefore it is necessary that the physical metallurgy, melting point temperature, solution of Laves phase, uniforming Nb segregation and relation of deformation with homogenization practice about the alloy ingots are studied here.

Materials and Experimental Procedure

Materials

The samples used in this study were cut from two Alloy 718 ingots of heat 943-31 and heat 043-27, produced by ShangHai Fifth Steel Plant, China, using VIM+VAR melting process. The size of samples is 12mm × 12mm × 12mm. The diameter of the ingots is 423mm. There were less contents of P, B • • • in heat 043-27 ingot. Their chemical compositions are seperately shown in Table1 and Table2.

Superalloys 718, 625, 706 and Various Derivatives
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Table 1 Heat 043-27 Chemical Composition (wt. %)

Nb	Ta	Mo	B	Al	Ti	Cu	Mn
5.25	<0.05	3.01	0.0043	0.60	1.04	0.02	0.02
C	Ni	Cr	Fe	Bi	Pb	Ag	Tl
0.035	52.42	18.70	18.23	<0.00001	0.00003	0.00001	0.00003
Co	Si	S	P	Sn	Mg	O	N
<0.05	0.15	0.005	0.0014	0.0009	0.0007	0.0004	0.0056

Table 2 Heat 943-31 Chemical Composition (wt. %)

Nb	Ta	Mo	B	Al	Ti	Cu	Mn
5.36	0.11	3.13	0.0052	0.60	1.00	0.068	<0.10
C	Ni	Cr	Fe	Bi	Pb	Ag	Tl
0.034	54.47	18.98	16.54	<0.00001	0.00005	0.00002	0.00003
Co	Si	S	P	Sn	Mg	O	N
0.099	0.14	0.001	0.0056	0.0032	0.0017	0.0003	0.0057

Procedure

A metallography and TEM coupled with the microprobe method were used to study the phases and dendritic segregation of Alloy 718 ingots. The initial melting point temperature was measured by Differential Thermal Analysis (DTA) and metallography methods. After heating the samples cut from the ingots at different temperatures the amount and morphology of Laves phase were examined by optical microscope, by which the curves of time of solutioning Laves phases with heating temperature can be obtained. After eliminating Laves phases "TAG" and microprobe methods were used to study the further decreasing of Nb segregation when the samples were continuously heated. A "TAG" method demands that samples are treated one hour at 870℃ after solutioning δ-Ni_3Nb phase. The large amount of γ'' precipitates in Nb rich regions and small amount of γ'' precipitates in Nb poor regions can be easily detected by optical metallography. The Nb rich regions appear dark (colour), the Nb poor regions appear white (colour) under optical microscope. The "TAG" treatment is very successful in delineating the Nb segregation.

Results and Discussion

The Physical Metallurgy of Alloy 718 Ingots

Figure 1 shows the optical metallographs of the edge and centre in heat 943-31 ingot. Except white MC precipitates with straight side, the other white islands are Nb rich Laves (TCP) phase. The Laves phase contains about 30 wt. % Nb and appears in the interdendritic areas. There are also a large amount of the long plate shape orthorhombic δ-Ni_3Nb phases around the Laves phases (Figure 2). This is confirmed by diffraction pattern (Figure 2d). The heat 043-27 ingot metallograph is similar to that of the heat 943-31 ingot. Figure 1 indicates that the edge of the ingot has much fewer Laves phases than the centre of it. This is because of the solidification of the Alloy 718 VAR melting, the edge of the ingot had much faster cooling speed than the centre. The faster cooling rate, the less segregation, the fewer Laves phases.

The Initial Melting Point Temperature of Alloy 718 Ingots

The DTA curve of heat 943-31 is shown in Figure 3. The DTA samples were cut from the centre of the ingots. It can be seen that the first intercept temperature on the DTA curve in Figure 3 is about 1167℃ which is considered to be the initial melting point temperature of the ingot. That is γ/Laves eutectic isotherm temperature. Figure 4 shows the microstructures of the samples kept at different temperatures for twenty minutes. The samples were cut from the

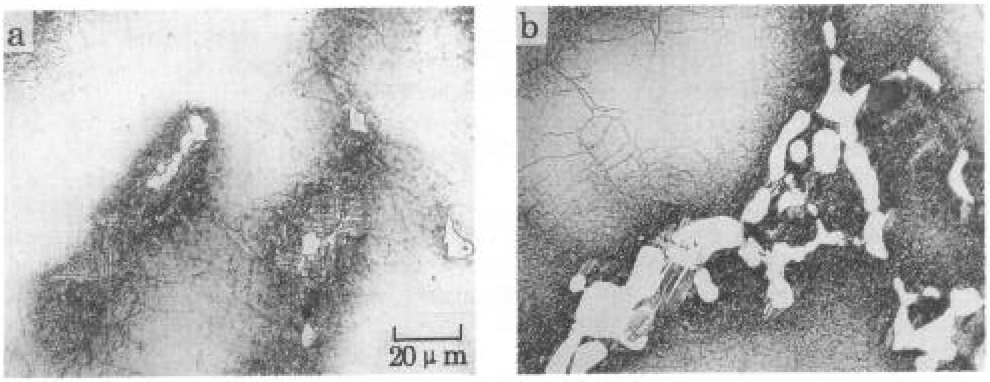

Fig. 1 Heat 943-31 metallograph a) edge and b) centre

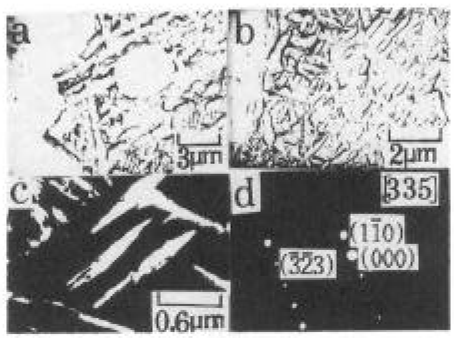

Fig. 2 Heat 943-31 TEM micrographs
a) and b) TEM replica micrographs
c) dark field d) diffraction pattern

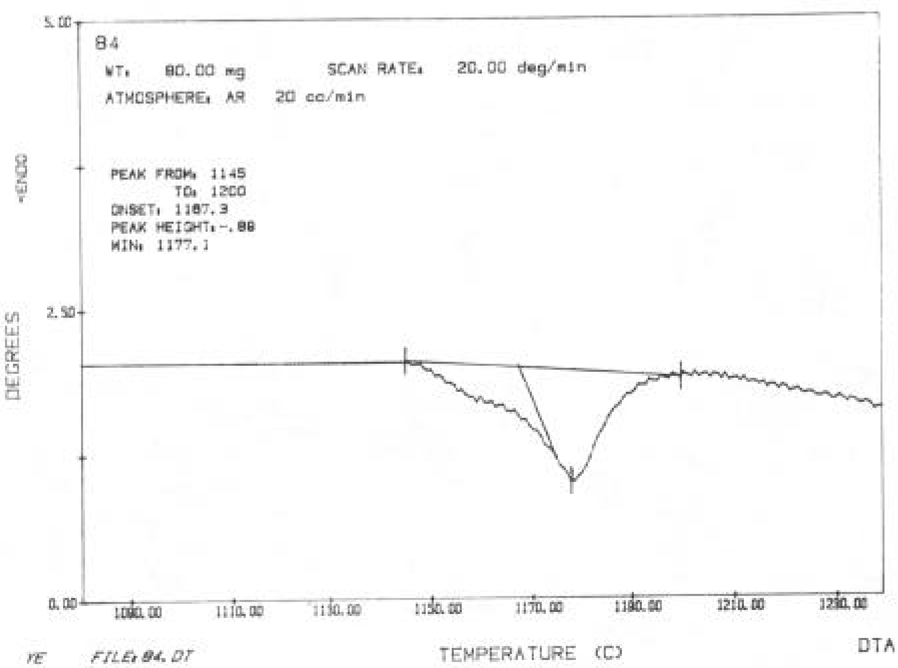

Fig. 3 DTA thermogram from heat 943-31

centre of the ingot. In the heating samples of 1175℃ and 1200℃ finer γ/Laves eutectic microstructure appear (Figure 4c,d), which demonstrates that the ingot is melting. However, the samples heated at 1145℃ and 1163 ℃ don't have this morphology. It suggests that the initial melting point temperature of the ingot is between 1163℃~1175℃. This result is consistent with that of the DTA method. Hence the initial melting point temperature of Alloy 718 ingots is about 1167℃. When the samples were first heated at 1160℃ for 13hrs, the Laves phases were eliminated, the melting point of the ingots rised to the temperature range of 1205℃~1215℃ (Figure 5). As the samples were heated at 1215 ℃ and 1230℃ (Figure 5 (c), (d)) porosities and finer γ/Laves eutectic microstructure were observed, which indicates the melting of the ingots. However when the samples heated at 1190℃ and 1205℃ (Figure 5(a), (b)) no porosities and γ/Laves eutectic microstructure have been found.

The Pattern Laves Solution

Table 3 and Figure 6 show the elements distribution of Alloy 718 ingots from dendrite to interdendritic regions. The Nb contents change from 2. 22wt. % to 32. 12wt. % in heat 943-31, from 3. 23wt. % to 31. 80. wt% in heat 043-27. It is noticeable that Nb segregation degree in the heat 943-31 ingot is different with that in the heat 043-27. Besides the Nb

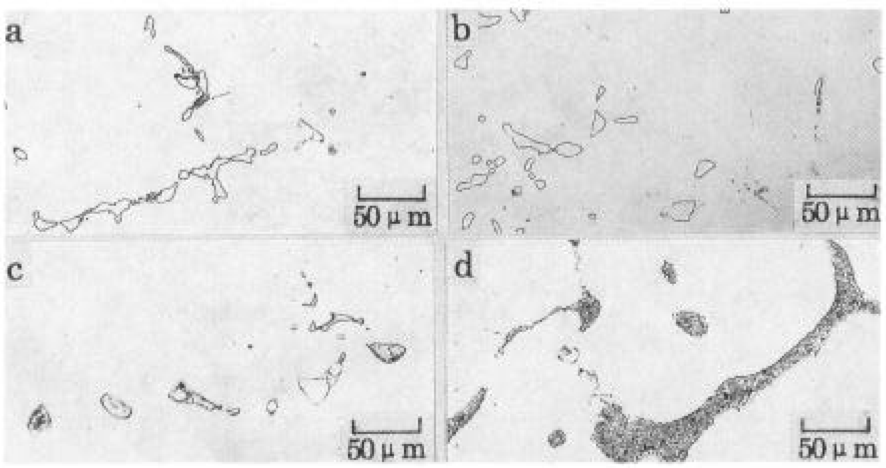

Fig. 4 Microstructure of Alloy 718 ingots at different temperature
a) 1145℃ b) 1163℃ c) 1175℃ d) 1200℃

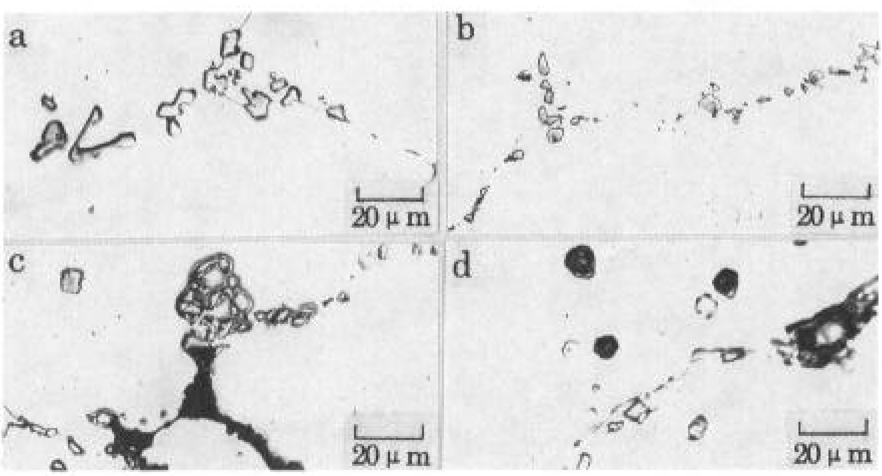

Fig. 5 After 1160℃ × 13hrs the microstructure of Alloy 718 ingots at different temperature
a) 1190℃ b) 1205℃ c) 1215℃ d) 1230℃

content difference in the dendritic centre, for example at the point of 30μm from the interdendritic regions the Nb content is 3.58wt.% in heat 943-31 but 5.98 wt.% in heat 043-27. The segregation degree in heat 043-27 is lighter than that in heat 943-31, the reason of which is that the impurity contents, such as P, Si • • • in heat 043-27, are smaller than that in heat 943-31, which will decrease the melting temperature range between liquidus phase boundary and solidus boundary[2][6], and hence the segregation during solidification in heat 043-27 is lighter than that in heat 943-31. Figure 7 shows the Laves solutioning of the alloy ingots hold in different times at different temperatures. There remain a few of white Laves phase islands in Figure 7(a),(c),(e),(g), and there is no Laves phase in Figure 7 (b),(d),(f),(h). According to results of Figure 7, the pattern of Laves phases solutioning of the alloy ingots was obtained, as shown in Figure 8, i.e.,

$$t = Ae^{-0.036T} \qquad (1)$$

Table 3 Elements Content in the Dendrite (wt.%)

Distance (μm)	Ti	Al	Cr	Fe	Ni	Mo	Nb
0	0.75	0.44	19.80	19.95	55.32	2.89	2.22*
	0.83	0.52	19.33	20.88	52.99	2.7	3.23**
30	0.70	0.44	20.16	19.96	55.45	2.88	2.69*
	0.96	0.51	19.23	20.20	53.74	2.87	3.2 **
60	0.73	0.51	20.31	20.42	55.15	2.84	2.43*
	1.21	0.49	19.22	19.92	52.80	3.07	4.48**
90	0.86	0.53	20.17	19.82	54.66	2.84	2.52*
	1.22	0.49	19.00	19.94	52.46	3.20	4.30**
120	0.91	0.53	20.09	19.22	54.32	2.99	3.58*
	1.30	0.51	18.53	19.03	53.65	3.50	5.98**
150	1.47	0.18	13.03	11.29	34.30	7.88	32.12*
	1.47	0.11	12.54	12.70	37.53	7.86	31.80**

*Heat943-31 **Heat043-27

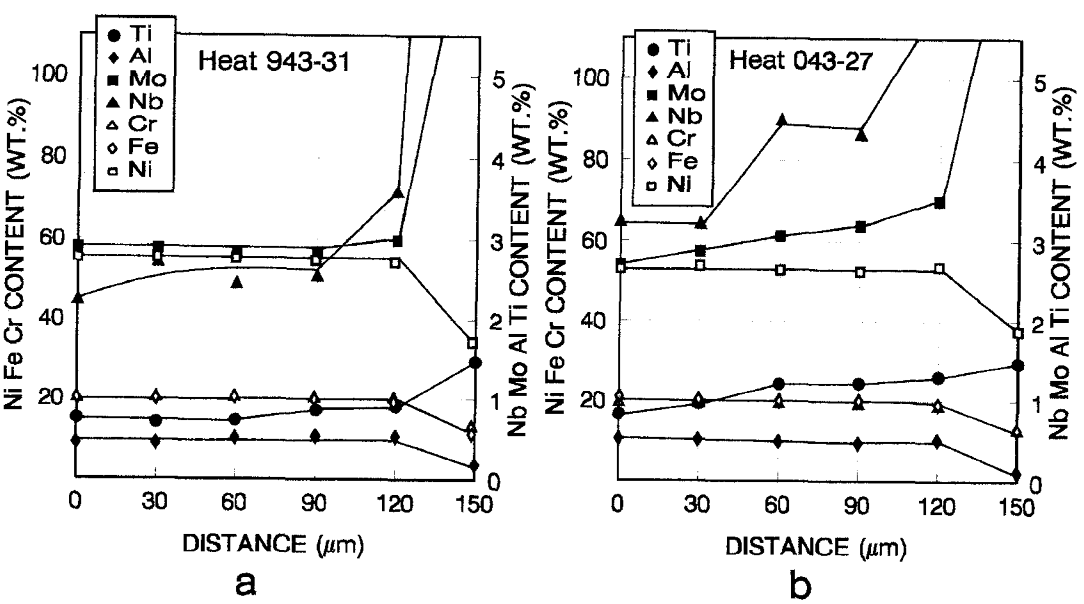

Fig. 6 Elements distribution from dendrite to interdendritic region
a) Heat 943-31 b) Heat 043-27

where t is the time of eliminating Laves (Hr), T is heating temperature (℃), the coefficient A is decided by the segregation degree of alloy ingots, $A = 1.30 \times 10^{19}$ for heat 943-31, $A = 1.642 \times 10^{19}$ for heat 043-27. The reason why the time of solutioning Laves phases in heat 043-27 is longer than that in heat 943-31 can be explained by Figure 6. According to Fick diffusion law:

$$J = -D \frac{\partial c}{\partial x} \tag{2}$$

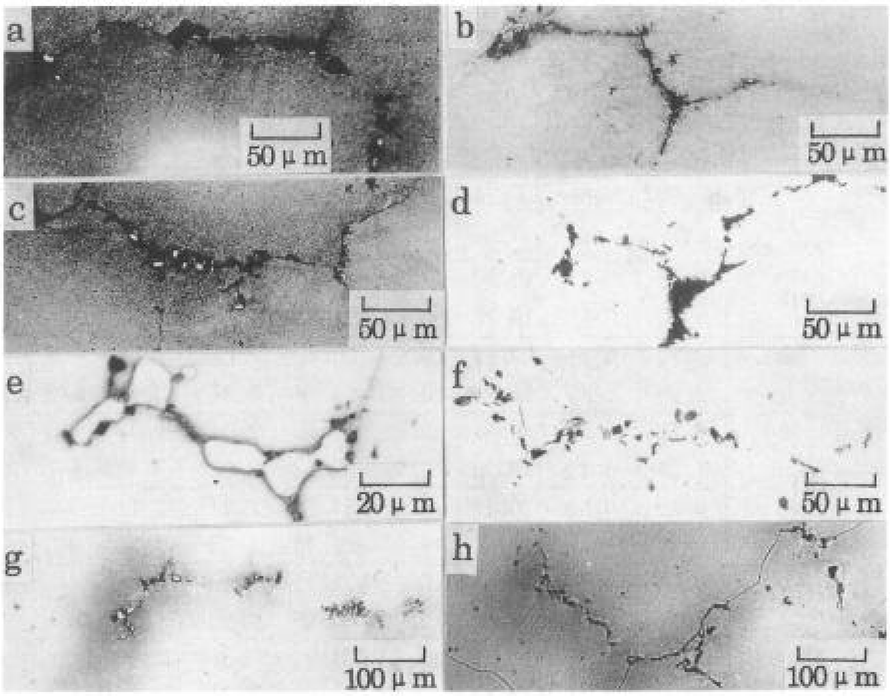

Fig. 7 Laves solutioning of Alloy 718 ingots at different temperature
a) 943-31 1120℃×31.5hrs
b) 943-31 1120℃×40.5hrs
c) 943-31 1140℃×17.5hrs
d) 943-31 1140℃×19.5hrs
e) 943-31 1160℃×8.5hrs
f) 943-31 1160℃×9.5hrs
g) 043-27 1160℃×9.5hrs
h) 043-27 1160℃×12hrs

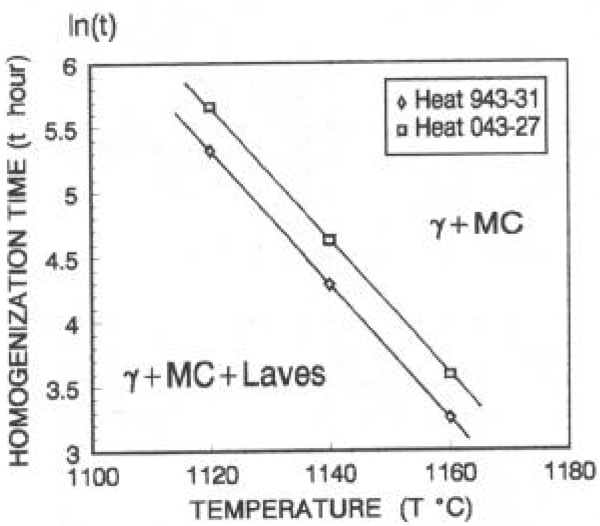

Fig. 8 Pattern of Alloy 718 ingots Laves solutioning

$$\frac{\partial c}{\partial x}(\text{heat } 043\text{-}27) < \frac{\partial c}{\partial x}(\text{heat } 943\text{-}31) \text{ (Figure 6)} \tag{3}$$

$$|J(043\text{-}27)| < |J(943\text{-}31)|. \tag{4}$$

Therefore Nb element diffusion in heat 943-31 ingot is faster than that in heat 043-27, elimination of Laves in the heat 943-31 is easier than that in the heat 043-27.

Further Homogenization of Nb of the Alloy Ingots after Elimination of Laves Phase

After the Laves was completely solutioned, the elements distribution in Alloy 718 ingots is shown in Figure 9. After heat treatment of 1160℃/12hrs for both heat 943-31 and 043-27 the Laves phases vanished, but Nb element segregation is still serious. It has been found that the segregation coefficient $K_{Nb}(943\text{-}31)=6.7/2.6=2.58$, $K_{Nb}(043\text{-}27)=6.18/3.23=1.91$ by assuming that $K=(C_{interdendritic})/(C_{dendritic})$. Therefore the homogenization of Nb in the alloy ingots is necessary after Laves phase solutioning. Figure 10 and 11 show the "TAG" examinating results of heat 943-31 and 043-27 ingots respectively after furtherly homogenizing. The white regions have low Nb content, the dark regions high Nb content. The Nb content in white regions can be as high as about 4.9 wt.%. When the white regions of the ingots are eliminated, the Nb segregation is obviously decreased. From the micrographs in Figure 10 and 11, it can be seen that there exist a few white regions in Figures 10 (a) 1160℃/107hrs, (b) 1180℃/63hrs and (d) 1200℃/52hrs, but no white regions in Figures 10 (c) 1180℃/100hrs, (e) 1200℃/80hrs, Figures 11 (a) 1160℃/57hrs, (b) 1180℃/30hrs and (c) 1200℃/20hrs. This indicates that homogenization of Nb for heat 043-27 is easier than for heat 943-31, as the segregation of Nb in heat 043-27 is lighter. The Nb contents in dendrite and interdendrite regions for both heats after the above further homogenization are shown in Figures 12, 13 and 14, respectively, measured by the microprobe. The Nb segregation coefficients, K_{Nb} in different homogenizing conditions are as follows, $K_{Nb}(943\text{-}31)=1.21$ $K_{Nb}(043\text{-}27)=1.08$ for 1160℃/107hrs (Figure 12), $K_{Nb}(043\text{-}27)=1.08$ for 1180℃/63hrs (Figure 13), $K_{Nb}(943\text{-}31)=1.08$ for 1180℃/100hrs (Figure 13), $K_{Nb}(943\text{-}31)=1.12$ $K_{Nb}(043\text{-}27)=1.08$ for 1200℃/80hrs (Figure 14). The above results indicate that the uniform of Nb distribution of Alloy 718 ingots can be obtained when the ingots were further homogenized after eliminating

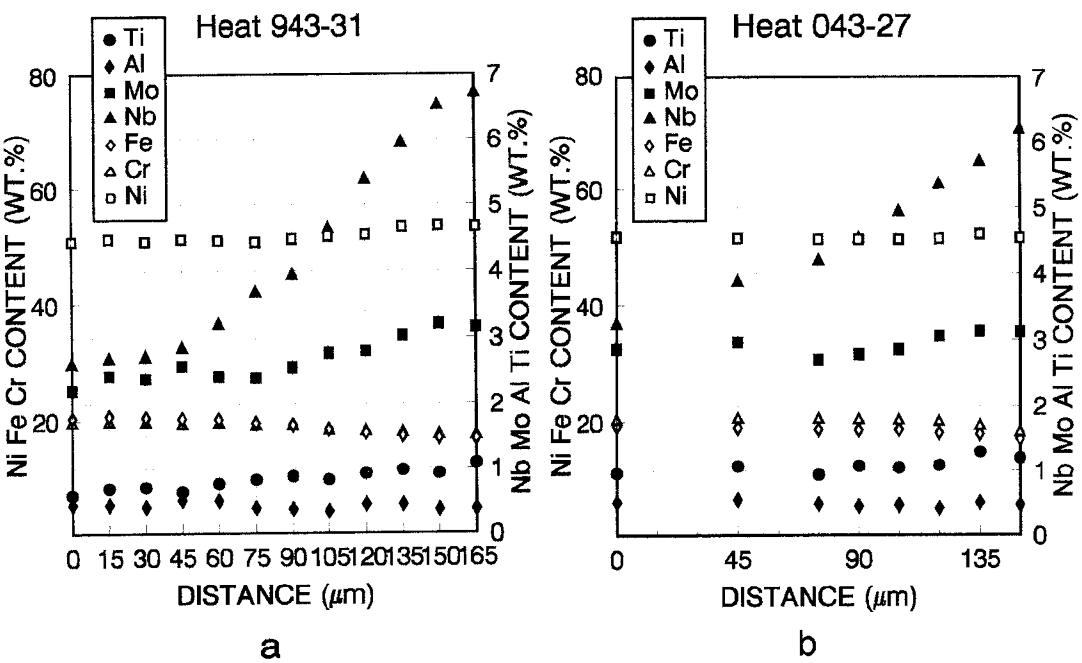

Fig. 9 Elements distribution in dendrite after 1160℃×12hrs
a) 943-31 b) 043-27

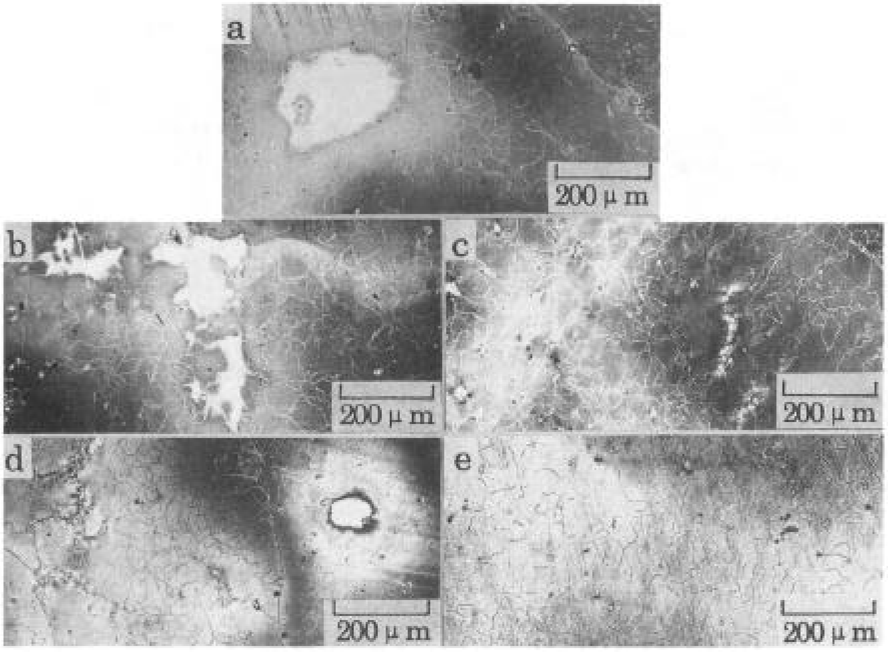

Fig. 10 Heat 943-31 "TAG" results
a)1160℃×107hrs b)1180℃×63hrs c)1180℃×100hrs
d)1200℃×52hrs e)1200℃×80hrs

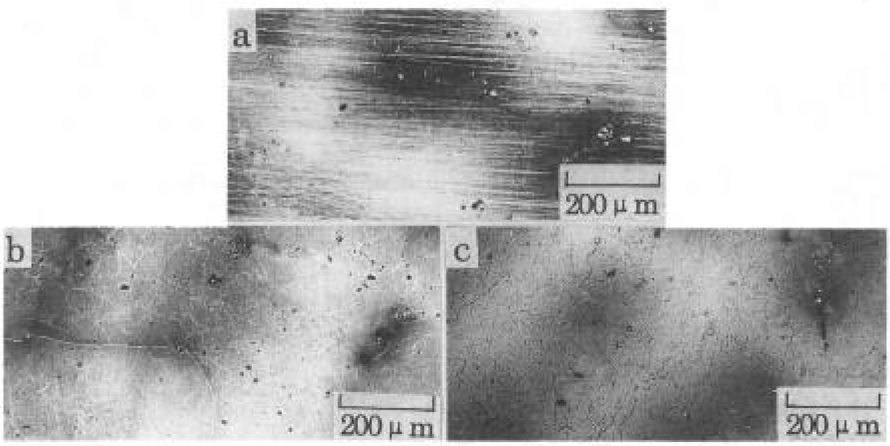

Fig. 11 Heat 043-27 "TAG" Results
a)1160℃×57hrs b)1180℃×30hrs c)1200℃×20hrs

Laves phases through 1180℃~1190℃×(70~100hr). The optimum homogenization treatment of Alloy 718 ingots is suggested to be 1150℃~1160℃ ×20hrs+1180℃~1190℃(70~100hrs). The accurate time (of 70~100hrs) should be chosen depending on the segregation degree of the ingots.

The Effect of Drawing Deformation On Alloy 718 Ingots Homogenization Practice

The "TAG" examination results of homogenized heat 043-27 ingot with drawn about 70% is shown in Figure 15. After heating 1160℃/80hrs, white regions can still be found out in the ingot by "TAG" method, up to 1160℃/100hrs the white regions completely vanish. But without the deformation, the homogenization treatment of 1160℃/57hrs can eliminate white regions for same heat of 043-27 (Figure 11). So the drawing deformation before the homogenization greatly increase the time of the uniforming ingots. The reason is that the drawing de-

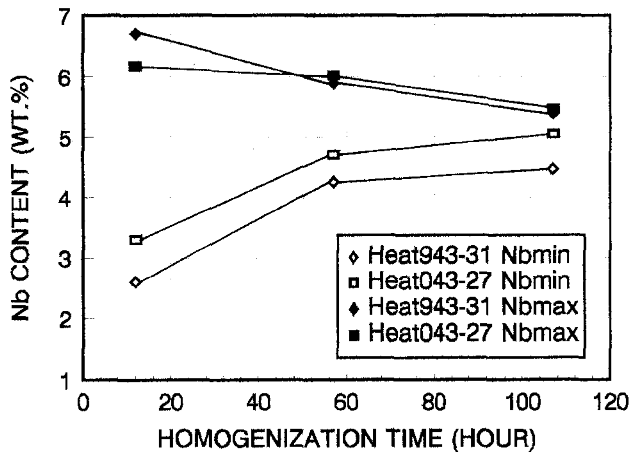

Fig. 12 Dendritic and interdendritic Nb content when 1160℃ heating

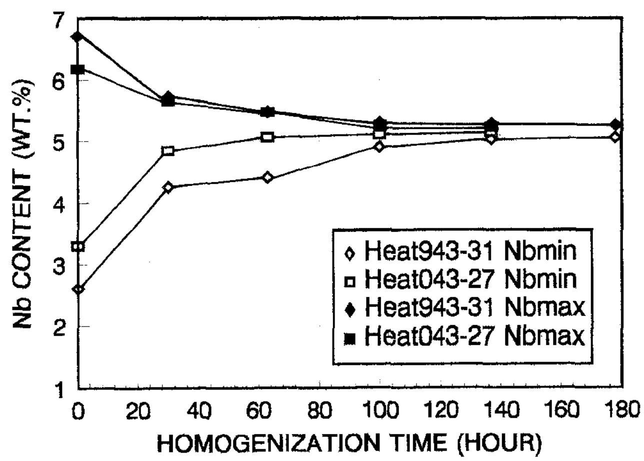

Fig. 13 Dendritic and interdendritic Nb content when 1180℃ heating

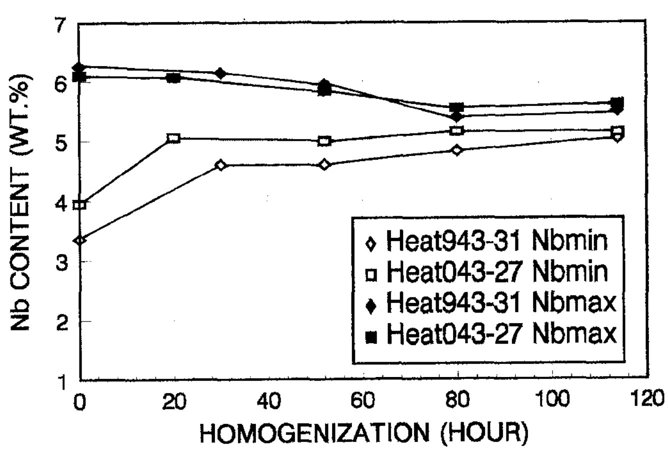

Fig. 14 Dendritic and interdendritic Nb content when 1200℃ heating

formation before the homogenization can cause Nb poor white regions concentrated (Figure 15 (a)), which made Nb diffusion distance increase and hence made the time of homogenization treatment increase. Therefore it is suggested that drawing deformation shouldn't be recommended before completely uniforming treatment.

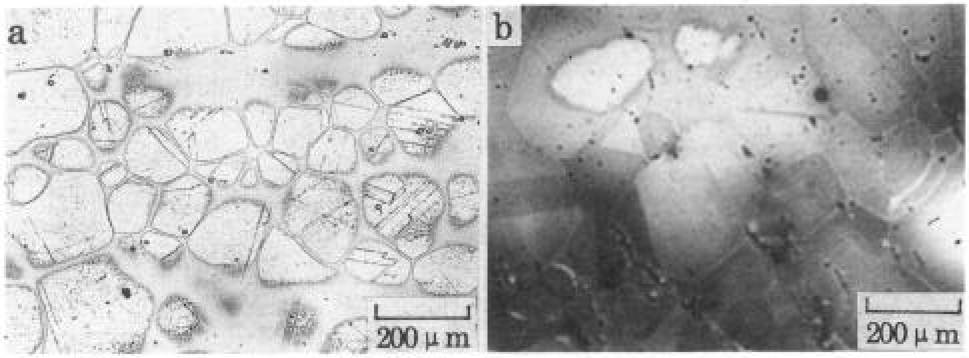

Fig. 15 "TAG" results of homogenized heat 043-27 with drawing deformation
a) 1160℃ × 20hrs + 80% • drawing
b) 1160℃ × 4.5hrs + 70% • drawing + 1160℃ × 80hrs

Conclusions

1. There is serious Nb segregation in Alloy 718 ingots. Nb rich Laves, NbC and δ phases segregate to the interdendritic regions, while the dendritic areas are Nb poor.
2. The initial melting temperature of Alloy 718 ingots is about 1167℃, the melting point temperature rises to around 1205℃ after eliminating Laves phases.
3. The relation between the eliminating Laves time and the heating temperature can be expressed in the following exponential function

$$t = A \cdot e^{-0.036T} \text{ (t-time(hr)) (T-temperature(℃))}$$

4. The optimum homogenization of heat treatment Alloy 718 ingots is recommended to be

$$1150℃ \sim 1160℃ \times 20\text{hrs} + 1180℃ \sim 1190℃ \times (70 \sim 100\text{hrs})$$

5. Before completely uniforming Alloy 718 ingots, the ingots should not be drawn to billets, as the non-uniform Nb distribution due to drawing is difficult to be eliminated.

References

1. John F. Radavich "The Physical Metallurgy of Cast and Wrougth Alloy 718", Superalloy 718, ed. E. A. Loria, (Pennsylvania, TMS, 1989), 229-240.
2. YaoXiao Zhu et al., "A New Way to Improve the Superalloys", Superalloys 1992, ed. S. D. Antolovich et al. (Pennsylvania, TMS, 1992), 145-154.
3. M. J. Cieslak et al., "The Solidification Metallurgy of Alloy 718 and Other Nb-Containing Superalloys", Superalloy 718, ed. E. A. Loria (Pennsylvania, TMS, 1989), 59-68.
4. G. K. Bouse "Application of a Modified Phase Diagram to the Production of Cast Alloy 718 Components", Superalloy 718, ed. E. A. Loria, (Pennsylvania, TMS, 1989), 69-77.
5. R. G. Carlson, J. F. Radavich "Microstructural Characterization of Cast 718", Superalloy 718 ed. E. A. Loria, (Pennsylvania, TMS, 1989), 79-95.
6. Zhu YaoXiao et al., "Superalloys with Low Segregation", Superalloys 1988, ed. S. Reichman et al. (Pennsylvania, TMS, 1988), 703-712.
7. J. M. Poole, K. R. Stultz and J. M. Manning "The Effect of Ingot Homogenization Practice on the Structure and Properties of Wrought Alloy 718", Superalloy 718, ed. E. A. Loria (Pennsylvania, TMS, 1989), 219-228.
8. D. F. Paulonis, J. M. Oblak and D. S. Duvall "Precipitation in Nickel-Base Alloy 718" Trans. of the ASM, Vol 62(1969).