THE EFFECT OF INGOT HOMOGENIZATION PRACTICE ON THE PROPERTIES OF WROUGHT ALLOY 718 AND STRUCTURE

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ABSTRACT

A controlled laboratory experiment was conducted to explore the effects of ingot homogenization practice on the properties of forged alloy 718. Five, 101 mm diameter alloy 718 VIM ingots were static cast, homogenized at various temperatures and hot forged to 25 mm squares. These forgings were evaluated by microstructural analysis, differential thermal analysis, room temperature tensile testing and 650°C stress rupture testing. Data from this investigation are supplemented by over 200 observations collected on 25 mm to 153 mm diameter hot rolled rounds during a time period when the VIM-VAR ingot homogenization practice was altered.

It was observed that the homogenization practice can have a profound effect on the macrostructure and microstructure of the as-cast and wrought products. Use of an improved step homogenization technique significantly reduces the number of rejections of wrought alloy 718 rounds due to banding, dendritic segregation or inadequate grain size control. The effect of the homogenization practice change on the room temperature tensile, 650°C temperature tensile and 650°C stress rupture results is relatively minor.

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INTRODUCTION

Current applications for alloy 718 are placing emphasis on improving the alloy's properties and quality. One approach to reach these goals is by thermomechanical processing (TMP). Here, the processing conditions are controlled with the objective to control the precipitation of delta phase [δ: Ni_3Nb], which, in turn, will have a significant impact on the tensile properties, stress rupture properties, low cycle fatigue properties and structure of the alloy(1,2). The key to successful TMP is the uniform precipitation of δ phase, and this is feasible only in a material free of niobium segregation. Unfortunately, cast alloy 718 suffers extensive segregation of niobium during solidification varying from about 4% (primary dendrite) to 30% (Laves phase), and is not amenable to TMP. This problem can be corrected by employing an ingot homogenization practice with the objective to solution the Laves phase and reduce the niobium gradient.

In earlier work(3), the effect of the ingot homogenization practice on VIM-VAR ingots was reviewed, and a two step homogenization practice was proposed. This practice consists of a pretreatment between 1149-1177°C followed by a 1204°C treatment. Here, the intent is to evaluate this practice relative to the structure and properties of wrought alloy 718.

PROCEDURE

Five, 15 kg lab ingots were prepared in a vacuum furnace and static cast into a 101 mm diameter graphite mold. A sample was cut for analysis, then the ingots were homogenized (TABLE I). Note that the niobium content was intentionally high to provoke a segregation problem. After homogenization, another sample was cut for evaluation, then all the ingots were heated to 1121°C/2 hour and forged to 25 mm squares in an identical fashion. All processed without incident except Ingot 4 which cracked and broke on the first hammer blow. Room temperature tensile testing (ASTM E8), combination smooth-notch bar stress rupture testing (ASTM E139: 650°C/760 MPa with K=3.6), and metallographic analysis were performed on the wrought bars in the direct aged and annealed plus aged temper. Differential thermal analysis (DTA) was performed on the annealed material.

In addition to the lab ingots, the effect of the homogenization practice on commercially produced 25-153 mm diameter hot rolled bars was investigated. The investigation centered on a random assortment of production orders for a period of eight months when the ingot homogenization practice was changed from 1191°C/50 hrs to a step practice consisting of 1163°C/24 hours plus 1204°C/72 hours.

TABLE I. Homogenization Practice and Chemistry (wt. %) of Lab Ingots.

Ingot	Homogenization	C	Fe	Si	Ni	Cr	Al	Ti	Mo	Nb
1	1150°C/8 hr AC	0.03	17.41	0.12	bal	17.85	0.73	1.02	3.06	5.43
2	1177°C/8 hr AC	0.02	17.13	0.14	bal	18.22	0.61	1.08	3.00	5.52
3	1191°C/8 hr AC	0.02	17.29	0.13	bal	18.00	0.64	1.08	3.01	5.60
4	1218°C/8 hr AC	0.02	17.57	0.14	bal	17.60	0.63	1.11	3.05	5.77
5	1177°C/2 hr + 1204°C/6 hr AC	0.02	17.57	0.13	bal	17.62	0.70	1.10	2.99	5.63

RESULTS

Lab Ingots

Macrostructure. The macrostructure of the as-cast lab ingots showed a typical structure with equiaxed and columnar regions. After homogenization, the macrostructure of Ingot 1 (1149°C) and Ingot 4 (1218°C) did not significantly change. The other ingots showed some obvious changes. Ingots 2 (1177°C) and 5 (1177°C + 1204°C) recrystallized to a comparatively fine grain structure whereas Ingot 3 showed grain growth. Seemingly, a fine grain structure can be realized when the initial homogenization temperature is below 1177°C---treatments over 1177°C (i.e., Laves solvus temperature) invoke grain growth.

Room Temperature Tensile and Stress Rupture Properties. The room temperature tensile results (TABLE II) indicate that there is little or no effect of the homogenization practice. The only obvious improvement is about a 70 MPa increase in strength in the direct aged tests when the homogenization treatment exceeded 1149°C. Likewise, the stress rupture results do not show any clear trends. Ingots with the higher homogenization temperatures (3 and 5) tend to have better stress rupture life and anneal plus aged rupture ductility. Also, the rupture ductility for Ingot 1 in the direct aged condition is higher than normally encountered which would suggest a poor ageing response.

TABLE II. Test Results for Lab Processed, Wrought alloy 718.

		Room Temperatue Tensile Properties				**650°C Stress Rupture**		
Ingot	**Temper**	**0.2% Yield (MPA)**	**Tensile (MPA)**	**Elong. (%)**	**Red. Area (%)**	**Life (hr)**	**Elong. (%)**	**Red. Area (%)**
1	A	1332.1	1494.1	13.0	31.0	82.8	12.7	31.4
	B	1462.4	1549.3	13.0	40.0	127.6	17.4	42.5
2	A	1299.7	1509.3	13.0	22.0	65.5	19.8	28.8
	B	1552.0	1615.5	10.0	32.0	54.4	13.6	49.9
3	A	1341.1	1498.3	13.0	36.0	139.9	24.9	41.8
	B	1515.5	1606.5	12.0	39.0	186.0	14.3	31.6
5	A	1283.9	1499.0	17.0	26.0	96.5	20.1	42.9
	B	1550.0	1623.8	11.0	41.0	151.0	10.2	19.4

Temper: A) 982°C/1hr AC plus treatment B.
B) 718°C/8hr furnace cool 38°C/hr 620°C/8 hr AC.

Microstructure. Phases observed in the as-cast cored structure include Laves, carbonitrides, delta (δ), and porosity. This is typical of cast alloy 718. In comparison to commercial material, the segregation in the 101 mm castings is minimal(2). Hence, results of this study will only reveal probable trends in commercial material.

For the cast and homogenized material, the homogenization practice has a profound impact on the structure. As the homogenization temperature is increased from 1150°C to 1191°C, the dendritic appearance fades, the degree of porosity increases (especially above 1177°C), the level of Laves phase is reduced and the carbide structure is altered (FIGURE 1). Here, only Ingots 1 and 2 showed the presence of some retained Laves phase; hence,

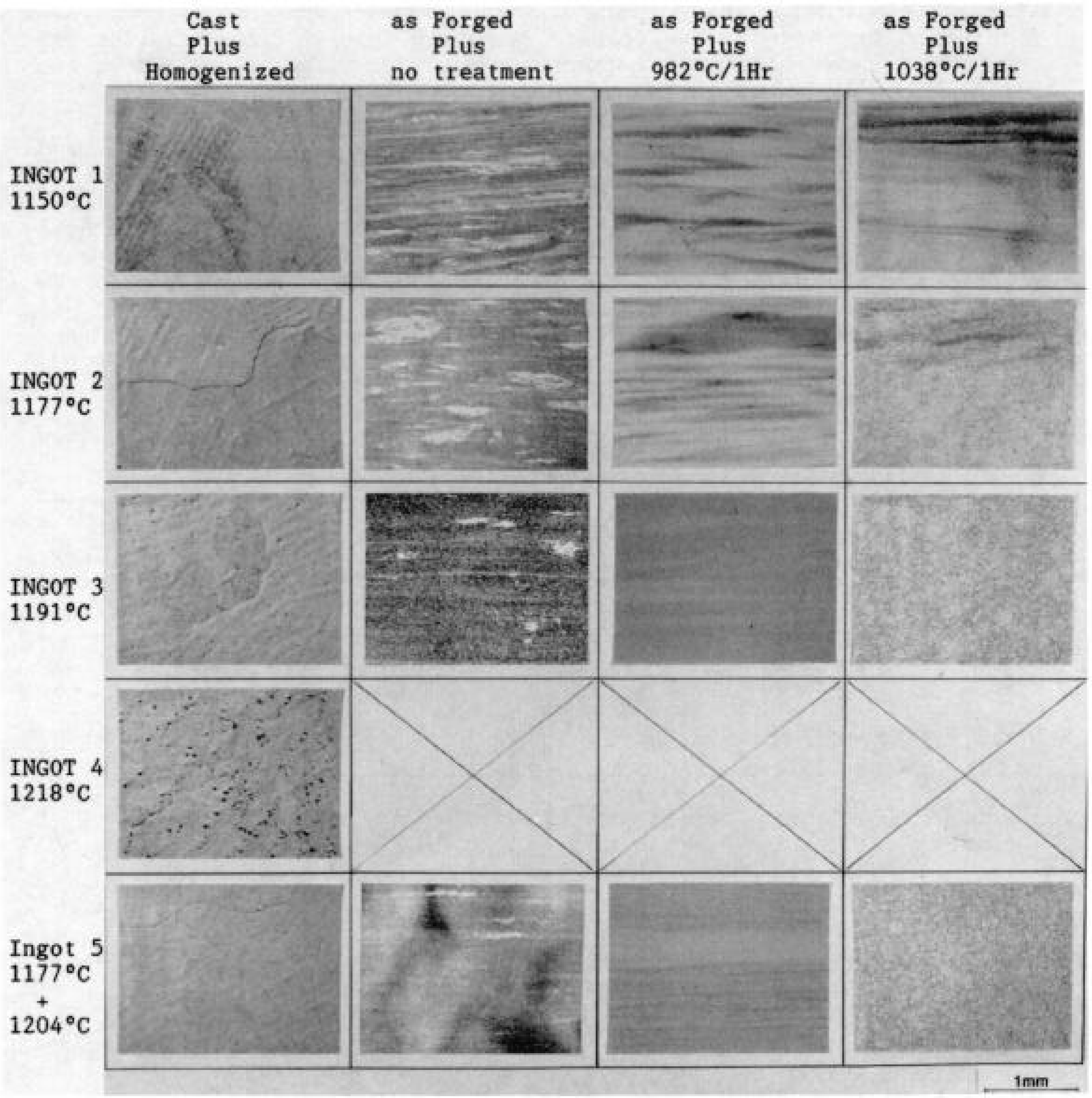

FIGURE 1: Photo Gallery depicting the relationship between structure, processing and homogenization treatment of the lab ingots.

either a longer treatment time or higher temperature is required to completely dissolve the Laves phase. Unfortunately, a higher, initial, homogenization temperature can be disastrous as illustrated by Ingot 4 (1218°C). This ingot showed a low melting point, niobium rich, grain boundary phase and could not be hot worked. Hence, the upper homogenization temperature is limited by incipient melting which was observed above 1191°C. (The degree of incipient melting observed in Ingot 3 was very slight and did not have any noticeable impact on the hot workability). Finally, there is a slight change in the carbide morphology from a small, clustered carbide (Ingot 1) to a large, randomly distributed carbide (Ingot 3). In the past this was attributed to Ostwald Ripening(3).

For wrought alloy 718, the change in the homogenization practice had a remarkable effect on the degree of banding and grain size. As the homogenization temperature is raised (1149-1191°C), the δ phase precipitation becomes more uniform which accounts for the improvement in the banding and grain size control (FIGURE 1). The best practice seemed to be the step practice (Ingot 5). The homogenization operation also influences the size and morphology of the primary MC carbides (FIGURE 2). Homogenizing below 1177°C promotes a finer, clustered carbide morphology in contrast to the large, random carbide morphology when the temperature exceeds 1177°C. For Ingot 5 (step practice), the carbide structure was similar to Ingot 2 (1177°C) suggesting that the low temperature treatment will stabilize the carbide size and morphology. The changes in the carbide size and distribution have been observed in earlier work(3,4).

Differential Thermal Analysis. DTA was performed with both a platinum and aluminium oxide reference on a Dupont 990 Thermal Analyzer using a 1600°C DTA cell, a Helium atmosphere and a 40°C/minute heating rate. Briefly, this analysis allows the researcher to determine the phase solvus temperature(s) by noting slope changes on the differential temperature trace. The solvus temperature can be equated to a particular phase with the aid of the alloy's TTT curve, corresponding metallographic analysis and

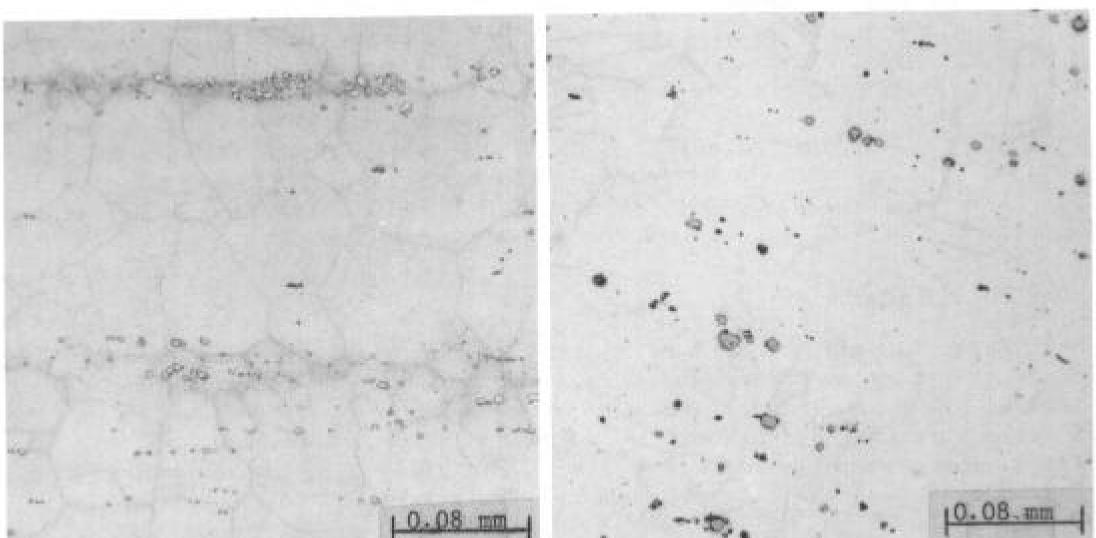

FIGURE 2: Carbide size and morphology for forged and 982°C annealed alloy 718 given a 1150°C homogenization treatment (Ingot 1: left) and 1191°C homogenization treatment (Ingot 3: right). Kallings Etch.

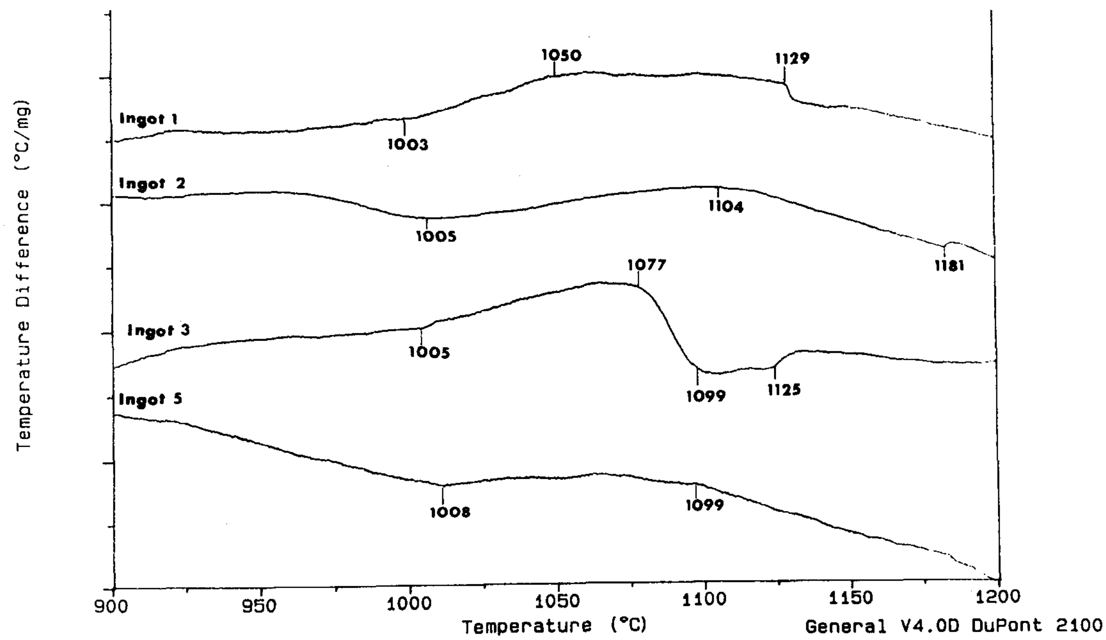

FIGURE 3: Differential Thermal Analysis curves for the wrought alloy 718 lab ingots. The δ phase solvus ranges from 1003-1129°C (Ingot 1), 1005-1104°C (Ingot 2), 1005-1125°C (Ingot 3) and 1008-1099°C (Ingot 5). The curve for Ingot 2 also shows a Laves phase solvus at 1181°C.

knowledge of the alloy's physical metallurgy. This is an excellent quantitative technique to evaluate the alloy 718 homogeneity. Non-homogeneous material will illustrate a wide δ solvus range due to the niobium gradient; hence, homogeneity may be evaluated by the range and the relative magnitude of the solvus temperatures. The results (FIGURE 3) show that the δ solvus range diminishes as the homogenization temperature is increased with the step practice yielding the best results. In addition to the δ solvus, the DTA analysis shows ' precipitation (550-640°C), ' solvus (770-803°C), " precipitation (698-724°C), " solvus (837°C), the alloy solidus (1243°C) and alloy liquidus (1341°C). Also, δ phase precipitation was observed between 927-961°C (Ingots 1, 2 and 3 only), and a Laves phase solvus near 1177°C (Ingots 1 [not shown on curve] and 2).

Commercial Production.

Here the effect of changing the homogenization practice from 1191°C/ 50 hours (practice 1) to 1163°C/24 hours plus 1204°C/72 hours (practice 2) on hot rolled, mill annealed bar products is reviewed by evaluating the alloy's structure and properties before and after the change. In this time frame, there were two different orders for alloy 718, the first for standard quality and the second for an oil field application.

Standard alloy 718. The averaged room temperature tensile, 650°C tensile and stress rupture properties for hot rolled, mill annealed and aged alloy 718 are presented in TABLE III. The change in the homogenization practice did not significantly affect the tensile properties but did slightly improve the stress rupture ductility.

TABLE III. Test Results for Commercially Produced alloy 718 Rounds.

Heat Treatment: 982°C/1 hr, 718°C/8 hr F.C. 38°C/hr 621°C AC.

Practice	Test Temp. (°C)	0.2% Yield (MPa)	Tensile (MPa)	Elong. (%)	Red. Area (%)	Life (hrs)	Elong. (%)
1	Room	1166.0	1389.3	19.6	38.4	----	----
47 obs.	650	968.7	1140.4	19.1	38.9	83.3	14.5
2	Room	1183.2	1405.9	18.2	37.4	----	----
73 obs.	650	1001.8	1173.5	20.4	40.3	84.2	19.2

TABLE IV. Room Temperature Tensile Data for alloy 718 (Oil Field Order).

Practice	Temper	0.2% Yield	Tensile	Elong.	Red. Area
1	A	351.6	777.8	59.1	64.0
102 obs.	B	857.0	1212.8	27.1	43.0
2	A	351.0	774.3	58.0	64.5
54 obs.	B	849.5	1198.0	26.9	44.2

Temper: A) 1023°C/1 hour water quench.
B) A + 788°C/8 hours air cool.

Production for the Oil Field Application. The specification for this order required a uniform grain size finer than ASTM 3 along with an annealed room temperature yield strength of 241-414 MPa and annealed plus aged properties of 827-1034 MPa yield strength, 1034 MPa minimum tensile strength, 20% minimum ductility and 25% minimum reduction of area. No high temperature tensile or stress rupture testing were specified.

As expected, the homogenization practice did not have any significant impact on the room temperature tensile properties (TABLE IV). However, it did have a dramatic effect on the structure. With the first homogenization practice, severe banding problems were encountered which violated the specification requirement for a uniform structure (FIGURE 4A). Efforts to correct the banding problem by increasing the rolling temperature (1010°C to 1038°C) solved the banding problem in many instances, but the grain size failed the ASTM 3 or finer requirement. The net result was a high rejection rate for these orders (FIGURE 5). Upon instituting the new homogenization practice, the structure improved considerably (FIGURE 4B) and problems with banding and coarse grain size vanished (FIGURE 5).

DISCUSSION

In earlier work(3), it was shown that ingot homogenization practice improves the gleeble hot ductility, room temperature tensile strength and the creep strength of cast alloy 718 with these same trends expected for the wrought material. It was concluded that a homogenization practice was required, and a step homogenization practice was proposed without a convincing demonstration of its value. Here, the task of this investigation was to build upon that work, and to define the best practice (defined as

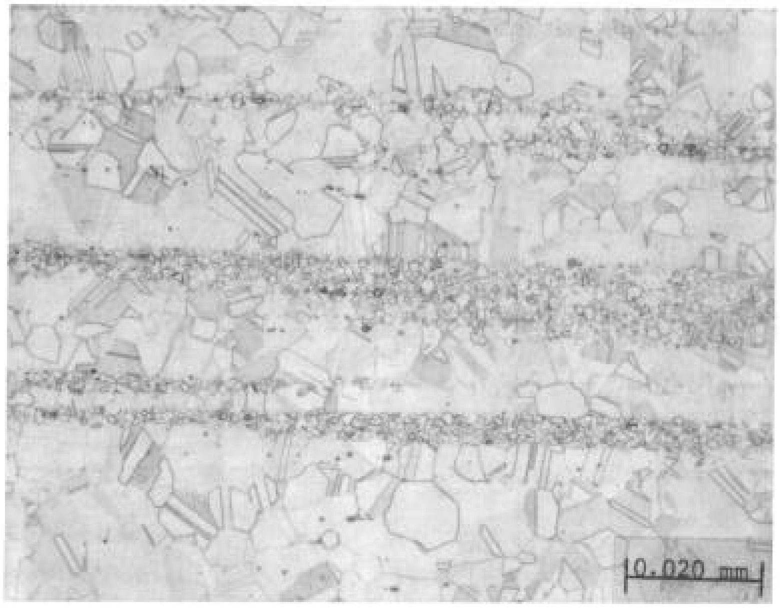

FIGURE 4A: Typical banded structure for wrought alloy 718 produced for the oil field application and using homogenization practice 1 (1191°C/50 hours).

FIGURE 4B: Typical structure for wrought alloy 718 produced for the oil field application and using homogenization practice 2 (1163°C/24 hours plus 1204°C/72 hours).

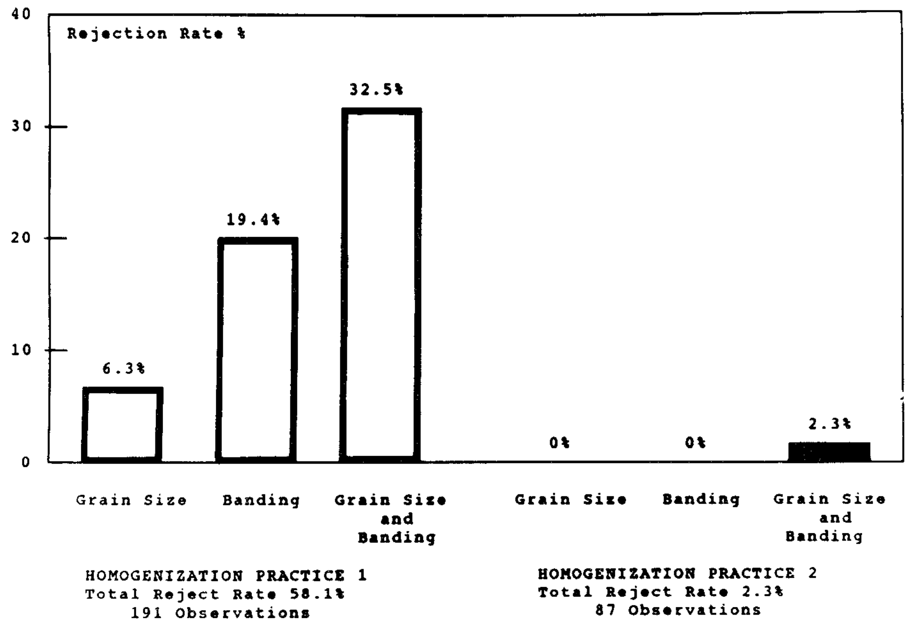

FIGURE 5. Rejection rate for oil field alloy 718 before and after the homogenization practice change.

the procedure which creates the most uniform niobium distribution) for wrought alloy 718. The intent is to produce a Laves free product that can be thermomechanically processed to form a uniform δ precipitate if desired.

Considering the lab investigation, Ingots 1 and 2 did not have an optimal homogenization practice as the Laves phase was not removed and banding still persists. Nor is the 1218°C practice (Ingot 4) suitable as the resultant incipient melting destroys the hot formability of the material. Thus the isothermal 1191°C practice (Ingot 3) and the 1177°C/2 hour plus 1204°C/6 hour practice (Ingot 5) appear best with the latter practice providing a slightly better grain size control and uniform δ solvus temperature. This is reinforced by the commercial production data. With the commercial data, there was no significant impact on the room or high temperature tensile properties, but there was a positive effect on the stress rupture ductility and microstructure. In fact, since this procedure was adopted as commercial practice over four years ago, the problems with banding have virtually disappeared.

The reasoning supporting a two step practice relative to an isothermal practice is relatively simple. For optimal Laves phase dissolution, the homogenization temperature should be between 1150-1177°C as higher temperatures provoke incipient melting and prolong the dissolution process. Unfortunately, this practice requires an excessive amount of time in order to produce an acceptable niobium distribution. This can be remedied by increasing the temperature which will enhance the niobium diffusion as it follows an Arrhenius-type law. Thus, a two step homogenization practice is recommended with the first step being 1150-1177°C and the second at 1204°C. Homogenization times will vary depending on the degree of segregation (chemistry, local solidification time), and may be varied accordingly.

Lastly, in prior work and in the lab study presented here, it was observed that the homogenization practice influences the size and morphology of the primary MC carbides. Basically, the carbide size increases as the homogenization temperature increases in the 1150-1191°C range due to Ostwald Ripening. The carbide morphology also changes from a clustered to a more uniform dispersion. These trends could also be observed, to a much lesser extent, on the mill production material. Apparently, the longer time at the higher temperature in the commercial material caused some coarsening of the finer, low temperature stabalized carbide such that the difference in the carbide morphology was relatively minor. However, by altering the homogenization time, some impact on the carbide size and morphology can be expected.

CONCLUSIONS

A step practice consisting of 1150-1177°C/24 hours plus 1204°C/72 hours is recommended for commercial VIM-VAR alloy 718. This procedure minimizes the degree of undesirable banding, increases the annealed and aged stress rupture ductility and improves the effectiveness of thermomechanical processing for structural control relative to a simple isothermal practice.

REFERENCES

(1) "Thermomechanical Processing of INCONEL 718 and its Effect on Properties," Processing and Properties of Advanced High Temperature Alloys, ed., S.M. Allen, R.M. Pelloux and R. Widmer (Metals Park, Ohio: American Society for Metals, 1986), 125-137.

(2) J.F. Radavich and W.H. Couts Jr., "Factors Affecting Delta Phase Precipitation and Growth at Hot Work Temperatures for Direct Aged INCO 718," Superalloys 1984, (Metals Park, Ohio: American Society for Metals, 1984), 497-507.

(3) J.M. Poole, "Homogenization of VIM-VAR INCONEL alloy 718," (Paper presented at the Ninth International Conference on Vacuum Metallurgy, San Diego, California, 11-15 April 1988).

(4) P. Siereveld, T. Kelly, G. Cole, R. Widmer and J.F. Radavich, "Effect of HIP Parameters on Fine Grain Cast alloy 718," Superalloys 1988, (Metals Park, Ohio: American Society for Metals, 1988), 459-468.