Effect of Heat Treatment on the Structure and Properties of Extruded *P/M* Alloy 718

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The structure and mechanical properties of alloy 718 prepared from argon atomized powder have been investigated for a wide range of extrusion ratios and temperatures. The tensile and stress-rupture properties of extruded bar are sensitive to heat treatment. Notch ductility can be conferred through appropriate combination of solution anneal and intermediate temperature age. Structural evaluation shows that such treatment provides a uniform grain structure together with a coarse γ'' precipitate dispersion and small amounts of δ Ni₃Cb phase at grain boundaries.

ALLOY 718 is a versatile high strength, weldable wrought nickel-base alloy which was developed for use at temperatures up to about 704° C.

The physical metallurgy of alloy 718 has been the subject of a number of investigations. A considerable amount of work was done initially to determine the phases precipitated during aging.¹⁻⁴ It is now firmly established that hardening is due to the formation of the metastable, disc-shaped γ'' .^{5,6} This phase, which is coherent with the matrix, has an ordered body centered tetragonal structure (DO_{22} type superlattice) with a c/a ratio of about 2.044. The γ'' phase forms on the cube planes of the matrix such that:

and $\binom{(001)_{\gamma''}}{[100]_{\gamma''}} / (001)_{\gamma}}{[100]_{\gamma''}}$

Upon prolonged aging the γ'' phase is replaced by the equilibrium orthorhombic δ phase having the same chemical composition (Ni₃Cb). The δ phase may form either by a cellular precipitation at grain boundaries or as intragranular plates. Although the major strengthening phase in alloy 718 is the γ'' phase, the work of Paulonis *et al*⁵ revealed that the ordered face centered cubic γ' phase also forms, but in smaller quantities.

The exceptionally high strength of alloy 718 has enabled the alloy to gain utilization in gas turbine engines as discs, blades and various structural shapes. For the latter application, rolling to close tolerance is a common fabrication method although limited to relatively simple shapes. Extrusion offers greater flexibility but the high strength of the material limits the extrusion ratio that can be employed and restricts the size of the extruded part.

The advent of powder metallurgy (P/M) technology has added a new dimension to the manufacture of superalloy shapes. Developments such as the "filledbillet" technique⁷ enable complex shapes having quite thin cross sections to be extruded. At the present time there is still very little information regarding the properties of extruded P/M superalloy products. The present investigation was undertaken to broaden the knowledge of P/M alloy properties by a study of the effect of heat treatment on the structure and properties of extruded P/M alloy 718.

EXPERIMENTAL PROCEDURE

Preparation of Powder

Four 45 kgm melts were individually vacuum induction melted using virgin raw materials and cast into graphite molds. Subsequently, the ingots were remelted in an atomization unit, poured into a preheated tundish from a tap temperature of 1482°C and atomized with argon. The -80 mesh fraction of the powder from each of the four melts was then cone blended to provide sufficient powder for extrusion and mechanical property evaluation. Precautions were observed during powder handling to insure freedom from contamination although all-inert handling was not used. Later a fifth melt (alloy 5) was prepared and atomized in like fashion. This batch of powder was not blended with the original four melts. This material was used for additional mechanical property evaluation and for much of the structural studies. The chemical analyses of each of the atomized powders is given in Table I.

Extrusion

Powder was packed into mild steel cans each fitted with an evacuation tube. Each can of powder was evacuated to about 30μ pressure and heated for about 2 h at 260°C prior to sealing. Extrusion to round bar was performed at 982, 1038 and 1093°C and at ratios of 9:1 to 30:1. In each case, flat faced dies were employed.

Heat Treatment

Following extrusion, samples of bar were solution annealed at temperatures from 954 to 1177 °C. Two aging treatments were examined:

Treatment A: 8 h/718°C, FC to 621°C and hold 10 h/AC. Treatment B: 4 h/843°C + 8 h/718°C, FC to 621°C and hold 10 h/AC.

Mechanical Property Evaluation

Room temperature and 649° C tensile data were obtained using specimens having a 0.635 cm diam and 2.54 cm gage length. Duplicate 649° C stress rupture tests were performed using combination notch/smooth

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Table I. Chemical Analysis of Individual Atomized Powder H
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Heat Number	С	Cr	Мо	Fe	Al	Ti	Cb	0	Ν	Ni
1	0.041	19.3	3.3	18.1	0.38	1.2	5.3	0.0079	0.0059	Balance
2	0.045	19.3	3.2	17.9	0.44	1.2	5.2	0.0110	0.0056	Balance
3	0.049	19.7	3.2	18.1	0.36	1.2	5.1	0.0098	0.0067	Balance
4	0.042	19.2	3.2	18.1	0.48	1.2	5.2	0.0071	0.0048	Balance
5	0.037	19.3	3.2	18.4	0.42	1.1	4.8	0.0075	0.0045	Balance

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Note: Powder batches 1 through 4 only were cone blended to provide master stock.

Table II.	Effect of Solution	Anneal	on Stress	Rupture	Properties of
	Bar Given	Aging T	reatment	A*	

Test Condition: 649°C and 689 MN/m ²									
	Extrusion Cond	itions		Exection					
Solution Anneal	Temperature, °C	Ratio	Life, h	Location					
1 h/954°C	1038	16:1	2.3	Notch					
			1.1	Notch					
1 h/954°C	1093	16:1	23.9	Notch					
			1.1	Notch					
1h/954°C	1093	22:1	8.8	Notch					
			4.7	Notch					
1 h/1066°C	1038	16:1	86.2	Notch					
			34.8						
1 h/1066°C	1093	16:1	2.0	Notch					
			5.1	Notch					
1 h/1066°C	1093	22:1	0.6	Notch					
			0.6	Notch					
1 h/1177°C	1038	15:1	199.7	Notch					
			127.2	Notch					
1 h/1177°C	1093	22:1	5.9	Notch					

specimens having 0.635 cm diam. A notch acuity factor, K_t , of 3.5 was employed.

Metallography

Samples for examination by optical microscopy were prepared using standard metallographic procedures. An immersion etchant consisting of 92 pct HC1-5 pct H_2SO_4 -3 pct HNO₃ was found to provide adequate attack for structural evaluation. This etchant was also used in the preparation of surface replicas for electron microscopy.

Thin foils for transmission electron microscopy were prepared using a dishing technique. Discs, approximately 0.025 to 0.038 cm thick were spark machined from test samples and electropolished in the DISA A-2 solution using a PTFE holder to provide a dished profile. Following dishing the discs were further electropolished in a 10 pct perchloric acidethyl alcohol mixture until a small perforation occurred. At this point polishing was discontinued and the disc washed prior to examination in the electron microscope.

RESULTS

Mechanical Properties

Stress Rupture Properties. The 649° C-689 MN/m² stress rupture properties are listed in Tables II and III. Extruded bar solution annealed 1 h at 954, 1066 or



Fig. 1-Effect of annealing temperature on grain size of extruded bar. 1 h anneal.

1177°C and aged according to treatment A were all notch brittle (Table II). For the 954°C solution anneal, rupture lives were relatively insensitive to extrusion temperature and ratio. Increasing the solution annealing temperature for bar extruded 16:1 at 1038°C significantly improved rupture life but the alloy remained notch brittle. There was no similar improvement in rupture life with annealing temperature for bar extruded 22:1 at 1093°C.

Notch ductility was obtained by the application of a $4 \text{ h}/843^{\circ}\text{C}$ intermediate temperature aging treatment (treatment B), though not for all solution annealing temperatures (Table III). A solution anneal of 1 h at 1066°C provided notch ductility for all extrusion conditions. Rupture lives ranged from 21 to 81 h. Notch brittleness was observed for bar solution annealed at 982°C and 1177°C. Annealing at 1121°C appears to be a borderline case.

Tensile Properties. The tensile properties of bar solution annealed 1 h at 954 to 1177 °C and aged according to treatment A are listed in Table IV. In general the yield and ultimate tensile strengths exhibited at room temperature and 649 °C are typical of those quoted for conventionally cast and wrought alloy 718. The tensile ductilities, however, are rather lower; particularly those for 649 °C. Annealing at 1066 °C increases the yield and ultimate tensile strength. This is due to more complete solutioning of the δ phase (Ni₃Cb) [see Microstructure].

The tensile properties of bar given aging treatment B are shown in Table V. The imposition of the intermediate temperature age (4 h/843°C) causes a reduction in alloy strength at both room temperature and 649°C. The tensile ductilities, however, are better than those exhibited for bar given aging treatment A(Table IV). For the range examined, the tensile properties are fairly insensitive to solution annealing temperature and extrusion condition,

Table III. Effect of Solution Anneal on Stress Rupture Properties of Bar Given Agin	ng Treatment B*
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	Extrusion Cond	itions				
Solution Anneal	Temperature, °C	Ratio	Life, h	Pct Elongation	Pct R.A.	Fracture Location
1 h/982°C	982	9:1	3.0	4.0	6.3	Notch
			3.1	5.0	5.4	Notch
1 h/982°C	982	22:1	9.0	3.0	4.0	Notch
			6.3	2.0	2.4	Notch
1 h/1066°C	982	9:1	25.6	5.0	8.2	Smooth Section
			21.0	3.0	6.2	Smooth Section
1 h/1066°C	1038	16:1	42.2	4.0	6.2	Smooth Section
			34.8	1.6	5.1	Smooth Section
1 h/1066°C	1093	22:1	46.6	3.0	4.8	Smooth Section
			52.0	6.0	8.2	Smooth Section
1 h/1066°C	1093	30:1	81.0	4.0	8.0	Smooth Section
			77.0 [†]			Notch
1 h/1121°C	1038	16:1	93.4	_		Notch
			99.0	5.0	4.8	Smooth Section
1 h/1121°C	1093	30:1	98.2	_	_	Notch
			72.8		-	Notch
1 h/1177°C	1038	16:1	75.8	0.8	0.8	Notch
			56.8	_	-	Notch

C, FC to 621°C, hold 10 h/AC.

[†]Determined to be improperly machined specimen.

Table IV. Tensile Properties of Extruded Bar Given Aging Treatment A^*

			Tensile Properties								
				Room Tem	perature		649°C				
	Extrusion Conditions		0.2 Pct Y.S	UTS	Pet		0.2 Pet VS	UTS	Pet		
Solution Anneal	Temperature, °C	Ratio	(MN/m^2)	(MN/m^2)	Elongation	Pct R.A.	(MN/m^2)	(MN/m^2)	Elongation	Pct R.A.	
1 h/954°C	1038	16:1	1250.5	1471.7	15	19	1039.7	1197.5	10	14	
1 h/954°C	1093	16:1	1198.2	1408.3	14	22	1022.5	1182.3	10	13	
1 h/954°C	1093	22:1	1187.1	1402.8	14	21	989.4	1113.4	10	11	
1 h/1066°C	1038	16:1	1329.1	1447.6	17	36	1077.6	1209.9	13	19	
1 h/1066°C	1093	16:1	1218.7	1465.5	15	25	1057.6	1206.4	11	15	
1 h/1066°C	1093	22:1	1290.5	1450.3	15	24	1052.1	1172.7	8	12	
1 h/1177°C	1038	16:1	1304.3	1464.8	22	37	1053.5	1187.1	16	22	
1 h/1177°C	1083	22:1	1191.9	1355.9	17	27	938.4	1064.5	22	27	

*Treatment A: 8 h/718°C, FC to 621°C, hold 10 h/AC.

Table V. Tensile Properties of Extruded Bar Given Aging Treatment B*

			Tensile Properties									
			Room Temperature						649°C			
	Extrusion Conditions		0.2 Pct Y.S.	UTS		Pct		0.2 Pet Y S	UTS	Pct		
Solution Anneal	Temperature, °C	Ŗatio	(MN/m^2)	(MN/m^2)	Elongation	Pct R.A.	(MN/m^2)	(MN/m^2)	Elongation	Pct R.A.		
1 h/1066°C	982	9:1			ND	_		923.9	1088.6	17	22	
1 h/1066°C	1038	16:1		_	ND		_	940.5	1149.9	13	15	
1 h/1066°C	1093	22:1		_	ND	-	_	917.1	1098.3	16	18	
1 h/1066°C	1093	30:1	1152.7	1434.5		14	20	1008.7	1150.6	13	15	
1 h/1121°C	1038	16:1	1109.3	1431.7		15	23	971.5	1152.7	18	27	
1 h/1121°C	1093	30:1	1100.3	1426.2		14	18	965.9	1157.5	17	22	
1 h/1177°C	1038	16:1	1115.5	1408.3		17	29	961.2	1138.9	18	26	

*Treatment B: 4 h/843°C + 8 h/718°C, FC to 621°C, hold 10 h/AC. ND = not determined.

Microstructure

Effect of Annealing Temperature. In the as-extruded state (e.g., 16:1 at 1038°C the grain size of P/M alloy is 10 μ m. The grain size is not significantly influenced by extrusion ratio or temperature in the range examined. A plot of grain size vs annealing temperature is shown in Fig. 1. There is a small increase in grain size upon annealing at 954 and 1066°C. This is due to solutioning of the δ phase as shown in Fig. 2(a) and (b). Annealing at 1177°C causes a more marked increase in grain size. The grain size however is nonuniform and exhibits a duplex size



(a)



(b)

Fig. 2—Micrographs illustrating structure of bar extruded 16:1 at 1038°C and annealed (a) 1 h/954°C, (b)1 h/1066°C and (c) 1 h/1177°C. Note presence of acicular Ni₃Cb for 954°C anneal.



(c)

distribution (Fig. 2(c)). The average grain size is 26 μ m.

Effect of Aging Treatment. Optical micrographs illustrating the effect of aging treatment on the structure of bar extruded 16:1 at 1038°C and annealed 1 h at 1066°C are compared in Fig. 3. It is evident that by employing the 4 h/843°C intermediate age (treatment B), a coarser general precipitate forms within the grains. Additionally, there is evidence of acicular δ phase at the grain boundaries.

Surface replication and thin foil electron microscopy were employed to examine the microstructure in more detail. Fig. 4 shows the microstructure of bar not given the intermediate temperature age and for which notch brittleness is experienced. Coarse MC type carbides are present throughout the grains. The grain boundaries are quite smooth and in only a few instances were small precipitates observed on them. The intergranular precipitate was identified by electron diffraction of thin foils as the body centered tetragonal γ'' phase (Fig. 5(a) and (b)). The dark field micrograph Fig. 5(c) shows that the γ'' precipitates continuously to the grain boundary. The γ'' formed by this treatment is typically 25 to 35 nM in diam and 7 to 12 nM thick.

The microstructure of material given the intermediate temperature age prior to the standard low temperature age (treatment B) is shown in Fig. 6. This structure confers notch ductility. Significant quantities of the acicular orthorhombic δ phase are formed at the grain boundaries producing an irregular morphology. The γ'' precipitate formed at 843°C is much coarser, typically 150 to 200 nM in diam and 15 to 30 nM thick, but does not precipitate immediately adjacent to the grain boundaries. During subsequent aging at 718°C and 621°C finer γ'' and γ' precipitates in these regions. The dark field electron micrograph shown in Fig. 7 was obtained using a 100 super-lattice reflection. For alloy 718, images due to both γ'' and γ' would be obtained.⁵ The γ'' would be seen edge on as lenticular particles. This





(b)

Fig. 3-Effect of an intermediate temperature age on the microstructure of bar extruded 16:1 at 1038°C. Samples annealed 1 h/1066°C and (a) aged using Treatment A (no intermediate temperature age) and (b) aged using Treatment B (with intermediate temperature age).

is evident for the coarse precipitates produced by the 843°C anneal. The finer precipitates are spherical which suggests these are predominantly γ' .

Higher solution annealing temperatures; *i.e.*, 1121 and 1177° reduce the amount of acicular δ phase that can form during aging at 843°C (Fig. 8). The irregular grain boundary morphology is less well developed and increased susceptibility to notch embrittlement is observed (Table III).

Fracture

Scanning electron micrographs illustrating the effect of heat treatment on the creep fracture characteristics at 649°C are shown in Fig. 9. In the notch brittle condition (aging treatment A) fracture occurs by intergranular crack nucleation and growth. As illustrated in Fig. 9(a) the fracture surface reveals predominantly smooth grain boundary facets indicating easy crack propagation.

By contrast, the 843°C aging treatment B which confers notch ductility, results in the fracture appearance illustrated in Fig. 9(b). Although crack propagation is still intergranular the fracture surface has a uniform ductile, dimpled appearance. The dimples



Fig. 4—Thin foil electron micrograph of extruded P/M Alloy 718 annealed 1 h/1066°C and aged according to Treatment A(no intermediate temperature age). Note smooth grain boundary morphology.



Fig. 5-Thin foil electron micrographs showing γ'' phase in extruded bar annealed 1 h/1066°C and aged according to Treatment A (no intermediate age). (a) Bright field, (b) diffraction pattern, (c) dark field of grain boundary region.







(c) Fig. 5-Continued.

are presumably due to the presence of δ phase at the grain boundaries which impede crack propagation and increase the work of fracture.

Deformation Characteristics

<u>Material Aged According to Treatment A</u>. The nature of creep deformation at 649° C for material aged according to treatment A is shown in Fig. 10. The deformation is coarse, planar and faulting is observed (Fig. 10(a)). Diffraction patterns (Fig. 10(c)) show streaks in $\langle 111 \rangle$ matrix directions. Note also the extra reflections at twinning positions (T) indicating that many of the faults are thick enough to be deformation twins. A dark field image of the twins obtained using a diffraction streak is shown in Fig. 10(b). Coarse planar deformation is a characteristic of nickel-base alloys containing fine γ'' (or γ') precipitates.^{5,8,9} Wilson⁹ has shown similar deformation structures for conventional wrought alloy 718 and has linked this to notch brittleness susceptibility.

Material Aged According to Treatment B. The application of an intermediate temperature age (4 h/ 843° C) allows more homogeneous deformation characteristics. Deformation twins are again observed but on a much refined scale. An example of the microtwinning in coarse γ'' is shown in Fig. 11(a). Again streaks are observed on diffraction patterns and can be used to image the microtwins in dark field (Fig. 11(b)). Dislocation loops are formed about the fine γ'' and γ' during aging at 718°C and 621°C (Fig. 12).







Fig. 6—Microstructure of P/M Alloy 718 annealed 1 h/1066°C and aged according to Treatment B (with intermediate temperature age). (a) Surface replica, (b) thin foil. Note coarse γ'' and presence of acicular δ phase (Ni₃Cb) at grain boundaries.



Fig. 7—Thin foil electron micrograph (dark field) showing γ'' and γ' precipitates at grain boundary region in bar annealed 1 h/1066°C and aged according to Treatment B (with intermediate temperature age).

This more homogeneous form of deformation is considered to contribute to attaining notch ductility.⁹

DISCUSSION

The results of this study show that P/M alloy 718 can be extruded over a fairly broad range of ratio and temperature ranging from 9:1 at 982°C to 30:1 at 1093°C and, therefore exhibits greater flexibility in extrusion than does conventional cast and wrought alloy 718 (Ref. 10). Conventional alloy 718 is sensitive to notch embrittlement unless final hot working is conducted at temperatures less than approximately



Fig. 8-Optical and electron micrographs showing effect of a 1 h/1177 °C solution anneal on the microstructure of P/M Alloy 718. Samples subsequently aged according to Treatment B.



Fig. 8-Continued.

Fig. 9-Scanning electron micrographs illustrating the fracture appearance of samples creep tested at 649°C. (a) Notch brittle (1 h/1066°C plus aging Treatment A) and (b) notch ductile (1 h/1066 °C plus aging Treatment B).

(a)

(c)

Fig. 10—Thin foil electron micrographs showing coarse planar deformation developed during creep at 649°C. (a) Bright field, (b) dark field, (c) diffraction pattern, [011] zone. Specimen heat treated 1 h/1066°C and aged according to Treatment A (no intermediate temperature age).

 982° C.¹⁰ In the present study it has been found that bar extruded at temperatures of 1038 and 1093°C can be rendered notch ductile though the application of an intermediate temperature (843°C) age prior to the commonly used aging treatment (8 h/718°C, FC to 621°C, hold 10 h/AC).

Three factors contribute to attaining notch ductility. The first is grain boundary morphology. The 843°C age allows small amounts of δ phase (Ni₃Cb) to develop at grain boundaries. The δ phase is beneficial because it provides an irregular grain boundary morphology. This impedes grain boundary sliding and as shown in Fig. 9(*b*) results in a more ductile fracture.

If solution annealed bar is not given the 843°C age

only fine γ'' and γ' precipitates form and these develop continuously to the grain boundary (Fig. 5(c)). The grain boundaries remain quite straight and provide easy paths for crack propagation (Fig. 9(a)). In this condition the material is notch brittle. Raymond¹¹ has noted that for conventional alloy 718, notch embrittlement can be avoided by preventing the formation of zones adjacent to the grain boundaries which are free of γ'' precipitates. This apparently is not a sufficient criterion for extruded P/M alloy 718. The nature of the grain boundary itself is a dominating factor.

The second factor affecting notch sensitivity is the prior solution annealing temperature. If the annealing temperature is too low (954 or $982^{\circ}C$) com-

Fig. 11-Thin foil electron micrographs showing fine planar deformation developed during creep at 649°C. (a) Bright field, (b) dark field. Specimen heat treated 1 h/1066°C and aged according to Treatment B (with intermediate temperature age).

Fig. 12-Bright field electron micrograph showing dislocation loops about fine γ' and possibly γ'' precipitates after creep at 649°C. Sample annealed 1 h/1066°C and aged according to Treatment *B* (with intermediate temperature age).

plete *initial* solutioning of δ phase is not obtained and notch embrittlement ensues even when the 843°C intermediate temperature age is applied. High solution anneals, 1121 and 1177°C also increase the susceptibility to notch embrittlement. For each of these cases the necessary grain boundary morphology is not properly developed when the 843°C intermediate temperature age is employed.

High solution annealing temperatures (1121 and 1177 °C), in addition to influencing the grain morphology that develops upon aging, also cause a nonuniform grain size distribution. Stroup and Heacox¹² have shown that a mixed grain size of this type can of itself lead to notch brittleness in conventional alloy 718.

They also observed that material which was notch sensitive could, in some cases, be rendered notch ductile if given a long time stress exposure (e.g., 500 h at 704°C and 345 MN/m² stress) prior to testing at 649°C. This overaging treatment causes a general coarsening of the γ'' precipitate and increases the amount of δ phase at grain boundaries. The presence of δ phase at grain boundaries in this case supports the contention that it is a necessary attribute in achieving notch ductility in extruded P/M alloy 718.

The third factor influencing notch sensitivity is the nature of the intragranular deformation. Wilson^{9,13} has employed this factor in discussing the time dependent notch embrittlement in both alloy 718 and Waspaloy sheet. The presence of a coarse γ'' (or γ' as in Waspaloy) leads to a more homogeneous deformation mode. This in turn inhibits crack nucleation. The present observations for P/M alloy 718 confirm the more homogeneous deformation character associated with the presence of coarse γ'' (developed by the 843°C intermediate temperature age). This homogeneous intragranular deformation mode undoubtedly plays a role in preventing notch embrittlement for P/M alloy 718. However, it is not apparently a sufficient criterion. The γ'' size and distribution developed by the 843°C age is essentially independent of solution annealing treatment. The mechanism of creep deformation, therefore, would be the same for all solution annealing treatments. If the nature of the intragranular deformation were the sole criterion influencing notch behavior, no grain size dependence (as affected by the solution anneal) would be expected.

CONCLUSIONS

1) The stress rupture properties of extruded P/Malloy 718 are sensitive to heat treatment. Notch ductility can be imparted through appropriate combination of solution anneal and intermediate temperature age. A heat treatment of 1 h/1066 $^{\circ}$ + 4 h/843 $^{\circ}$ + 8 h/718 $^{\circ}$, FC to 621 $^{\circ}$, hold 10 h/AC has been found satisfactory.

2) A uniform grain size in combination with coarse γ'' precipitates and small amounts of δ phase (Ni₃Cb) at grain boundaries appear to be necessary for maintaining notch ductility in extruded P/M alloy 718.

3) Thin foil electron micrographs reveal that an intermediate temperature aging treatment significantly modifies the deformation characteristics during creep and in tensile tests. Fine scale microtwinning of coarse γ'' coupled with dislocation looping about fine γ'' and γ' develops. This leads to a more homogeneous deformation and contributes to attaining notch ductility.

4) Notch ductility has been demonstrated in P/M alloy 718 bar extruded at 1038°C and 1093°C. This is in contrast to conventional cast and wrought alloy 718 for which finish hot working must be conducted at temperatures less than approximately 982°C if notch ductility is to be achieved.

5) P/M alloy 718 can be extruded over a broad range of temperature and extrusion ratio and in this respect exhibits greater flexibility than conventionally cast alloy 718.

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