THERMOMECHANICAL PROCESSING

OF P/M ALLOY 718

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Summary

The use of powder metallurgy technology in the production of Alloy 718 is a relatively recent event. Although considerable work has been done on the effects of various processing parameters on the structure and properties of cast and wrought Alloy 718, little is known about these effects in P/M Alloy 718. This study was undertaken to investigate the structural changes and mechanical property behavior induced in P/M Alloy 718 by thermomechanical processing. Argon atomized powder was consolidated via the CAP^{R} process and subjected to various hot working procedures and heat treatments. Tensile and stress-rupture testing as well as optical and electron microscopy and x-ray analysis were used to evaluate the heat treated material. It was found that P/M alloy 718 exhibits the same phases as its conventionally cast and wrought counterpart after similar solution treatments; however, both precipitate and second phase particles are finer and more uniformly dispersed in the powder material. The recrystallized structure of P/M Alloy 718 maintains a finer grain size at a given solution temperature, i.e., grain coarsening does not occur as readily as in wrought Alloy 718. Finally, P/M Alloy 718 demonstrates equivalent mechanical properties to the conventional material, indicating that a useful product with a fine uniform structure can be produced through powder metallurgy techniques.

Introduction

Cast and wrought Alloy 718 continues to be used in greater volume and for more applications with the passing of time. Performance demands have required modifications of the chemistry and production methods to meet high quality requirements. Numerous refinements have been made in the melting, remelting and processing of this alloy to finished parts, but the quest for even more improvement continues. One of the new approaches for higher quality involves the manufacture of Alloy 718 by powder metallurgy techniques.

While powder metallurgy techniques have traditionally been applied to more complex alloys which are otherwise difficult to manufacture because of alloy content and/or segregation, the development of more economical powder manufacturing methods have enabled the use of P/M technology for conventional superalloy production. The beneficial effects expected in Alloy 718 produced via powder metallurgy are superior cleanliness, elimination of segregation, finer grain size, and a finer and more uniform distribution of second phase particles. These characteristics should provide superior mechanical property levels, especially in terms of low cycle fatigue strength(1). This study was undertaken to investigate the structural behavior of P/M Alloy 718 when subjected to thermomechanical processing, and to relate structural changes to mechanical property behavior.

Experimental Procedure

Material Processing

Several heats of Alloy 718 were vacuum induction melted using remelt stock, and argon atomized to produce powder via the Commercial Rapid Solidification (CRS) process. CRS powder manufacturing methods take advantage of rapid solidification techniques to produce powders exhibiting exceptional microstructural uniformity and consistency. After atomization, the heats were screened to -80 mesh and blended in a vacuum blender/dryer. Initially, the blender serves to thoroughly mix the powder from the various heats. After mixing, during the initial phase of CAP (Consolidation by Atmospheric Pressure) processing, the blender serves as a vehicle by which a boric acid additive is evenly applied to each powder particle. This additive is designed to enhance diffusion and bonding during the subsequent consolidation operation.

The chemical composition of the powder blend, shown in Table I, was consistent with Alloy 718 chemistry specifications. Oxygen and nitrogen levels on the blend lot were low at 90 and 75 parts per million, respectively. Powder cleanliness was verified via water elutriation, a method by which contaminants of lower density are separated from the powder particles on the basis of specific gravity. The overall blend lot data indicated a contaminant level of 18.1 particles per kilogram (8.2 particles/pound), which compares favorably with a current average industry standard of 55 particles per kilogram (25 particles/pound). After powder inspection and characterization, the powder was consolidated into preforms. Powder was loaded into six inch diameter cylindrical molds made of borosilicate glass. Molds were attached to a vacuum degassing system and degassed to an outgassing rate of 0.4 microns per minute. Acetylene torches were used to soften the glass tubulation at the bottleneck so that the pressure differential drew the neck in and created an airtight seal. The encapsulated powder was then subjected to a consolidation cycle of 20 hours at 1227°C (2240°F) in a conventional air atmosphere furnace, followed by furnace cooling to approximately 760°C (1400°F) and then air cooling. The billets consolidated to 99 percent of theoretical density, and measured 12.2 cm (4.8 inches) in diameter by 85.1 cm (33.5 inches) long.

After grinding to condition the surface, billets were soaked at 1093°C (2000°F) and rolled a total of 78% to 5.7 cm (2.25 inch) diameter bar, using several reheats and

individual passes of from five to ten percent reduction per pass. Finish temperatures of bars off the mill were determined to be approximately 927°C (1700°F) using an optical pyrometer, although subsequent structural studies indicate that actual temperatures throughout the bars may have been higher.

Table I. Chemical Composition of P/M Alloy 718 (Wt. %)

Blend	C	S	Cr	Ni	Mo	Al	Ti	Cb	Fe
V169-B	.04	.003	18.61	53.35	3.02	.50	0.92	5.45	18.10
AMS5662									
min.	-	-	17.00	50.00	2.80	.20	0.65	4.75	bal.
max.	.08	.015	21.00	55.00	3.30	.80	1.15	5.50	

Testing and Evaluation

A two hour gradient bar study was conducted on the as-rolled material in order to explore the structural changes within a temperature range of 788°C to 1093°C (1450°F to 2000°F). Based on this study, temperatures of 871°C, 927°C, 982°C and 1038°C (1600°F, 1700°F, 1800°F, and 1900°F) were selected as the solution temperatures to heat treat the as-rolled material for further evaluation. Solution treatment was, in each case, followed by the usual low temperature age, consisting of 718°C (1325°F)/8 hours/cool at 56°C (100°F) per hour to 621°C (1150°F)/8 hours/air cool. It was felt that the material would normally be exposed to this temperature range through processing, cooling or isothermal heat treatments. Within this range, wrought 718 has been shown to precipitate delta, gamma double prime, Laves, and/or gamma prime phases through thermomechanical processing.[2]

Fifteen centimeter (6.0 inch) sections of 5.7 cm (2.25 inch) diameter bar were solution treated for two hours at the selected solution temperatures, rapid air cooled, and then aged as described above. Structural analyses were conducted on samples of material in the as-consolidated, as-rolled, and as-heat treated conditions. Standard optical metallographic techniques were used to observe grain size and structural characteristics on the light microscope. Samples were also examined on the scanning electron microscope (SEM) after electrolytic polishing in a 20% sulfuric-methanol solution. Residues were extracted from samples of the material in each condition via anodic dissolution. After grinding the surfaces to remove any oxide, the samples were immersed in a 10% hydrochloric-methanol solution for extraction of carbides, delta and Laves phase; a solution of 1% ammonium sulfate-1% citric acid in water was employed for extraction of gamma prime and gamma double prime precipitates. Residues were analyzed using a General Electric diffractometer using Cu K α radiation.

Mechanical property evaluation of the heat treated Alloy 718 material consisted of room temperature and 649°C (1200°F) tensile tests and stress-rupture tests at 649°C (1200°F) and 759 MPa (110 ksi). For each heat treated condition, two cylindrical tensile specimens with a 0.64 cm (0.252 inch) diameter and a 2.54 cm (1.000 inch) gage length were tested at each temperature and two stress-rupture specimens, 0.406 cm (0.160 inches) in diameter with a 1.65 cm (0.650 inch) gage length were tested. All specimens were oriented in the longitudinal direction. Tensile tests were conducted on an Instron tensile machine using a strain rate of 0.005 - 0.050 inches/inch/minute. Three-zone Arcweld creep testing machines, controllable to plus or minus 1.7°C (3.0°F), were used for stress rupture tests. Structural evaluations were carried out on selected test samples.

Results

Microstructural Characterization

Figure 1(a) shows the structure of the argon atomized Alloy 718 powder as observed on the SEM. Spherical particles with surface skins and satellites, typical of gas atomized powder are observed. The structure of consolidated material can be seen in Figure 1(b). The as-CAP structure shows uniform equiaxed grains of ASTM grain size #6 with carbides and nitrides of a finer nature than those seen in wrought 718 evenly dispersed throughout. A lamellar Ni_3Cb precipitate is evident in the grain boundaries of this material. Also present in the grain boundaries are many much smaller particles with a plate-like morphology, and some areas of fine round precipitates. It is evident that the consolidated material precipitated various phases during the cooling process.

Rolling the consolidated material at 1038°C (1900°F) resolutions these precipitates leaving the grain boundaries free of precipitation, as seen in Figure 1(c). However, the high magnification SEM micrographs demonstrate that the cooling of the rolled bar results in precipitation of a very fine gamma double prime and/or gamma prime precipitate. The as-rolled grain size is ASTM #10.5. Some duplexing of grains was observed near the edge of the as-rolled bar, indicating possible non-uniformity of working. As in the consolidated material, many fine and widely dispersed second phase particles are present.

The grain size is unaffected by solution treatment at 871°C (1600°F), as evidenced by Figure 1(d). Acicular delta phase is present in the grain boundaries, while the grain matrix displays numerous disk-shaped gamma double prime particles. Any fine gamma prime particles which might have precipitated during the aging treatment would be difficult to detect because of the large amount of the gamma double prime phase present.

After solution treatment at 927°C (1700°F), more extensive delta phase is present in the grain boundaries, although the disk shaped gamma double prime is not observed. These phenomena are demonstrated in Figure 2(a). Grain size is again unaffected by this heat treatment. The SEM micrographs show fine gamma double prime precipitation in the grain matrix due to the aging process.

Solution treatment of the rolled material at 982°C (1800°F) results in the structure shown in Figure 2(b). In this condition, considerably less grain boundary delta phase is present. Grain coarsening is evident in the areas that are free of delta, while other grains stay fine because of the presence of delta at the boundaries. The average grain size has coarsened to ASTM #9, still fine relative to the grain size of similarly treated wrought Alloy 718. Delta phase platelets appear more discrete in this sample than in the sample solution treated at 927°C (1700°F).

As observed in Figure 2(c), when the solution temperature is raised to 1038°C (1900°F), all delta phase is dissolved. Grain growth occurs to a grain size of ASTM #7, which is comparable to the response of conventional material solutioned in the 927°C (1700°F) range. The material exhibits a very fine gamma prime or gamma double prime precipitate in the grains after aging, which is finer than those detected in the samples solution treated at the lower temperatures.

The overall structural trends with changing solution temperature are in agreement with the results of the gradient bar study. This study indicated that at approximately 788°C (1450°F), an easily detectable matrix gamma double prime precipitate is present, as shown in Figure 3, and delta phase has formed at the grain boundaries. Above 871°C (1600°F), gamma double prime is no longer observed in the matrix. Also, as observed in Figure 4, delta phase is gradually taken into solution as solution temperature increases from 968°C (1775°F) to 1010°C (1850°F), and grain growth occurs accordingly.

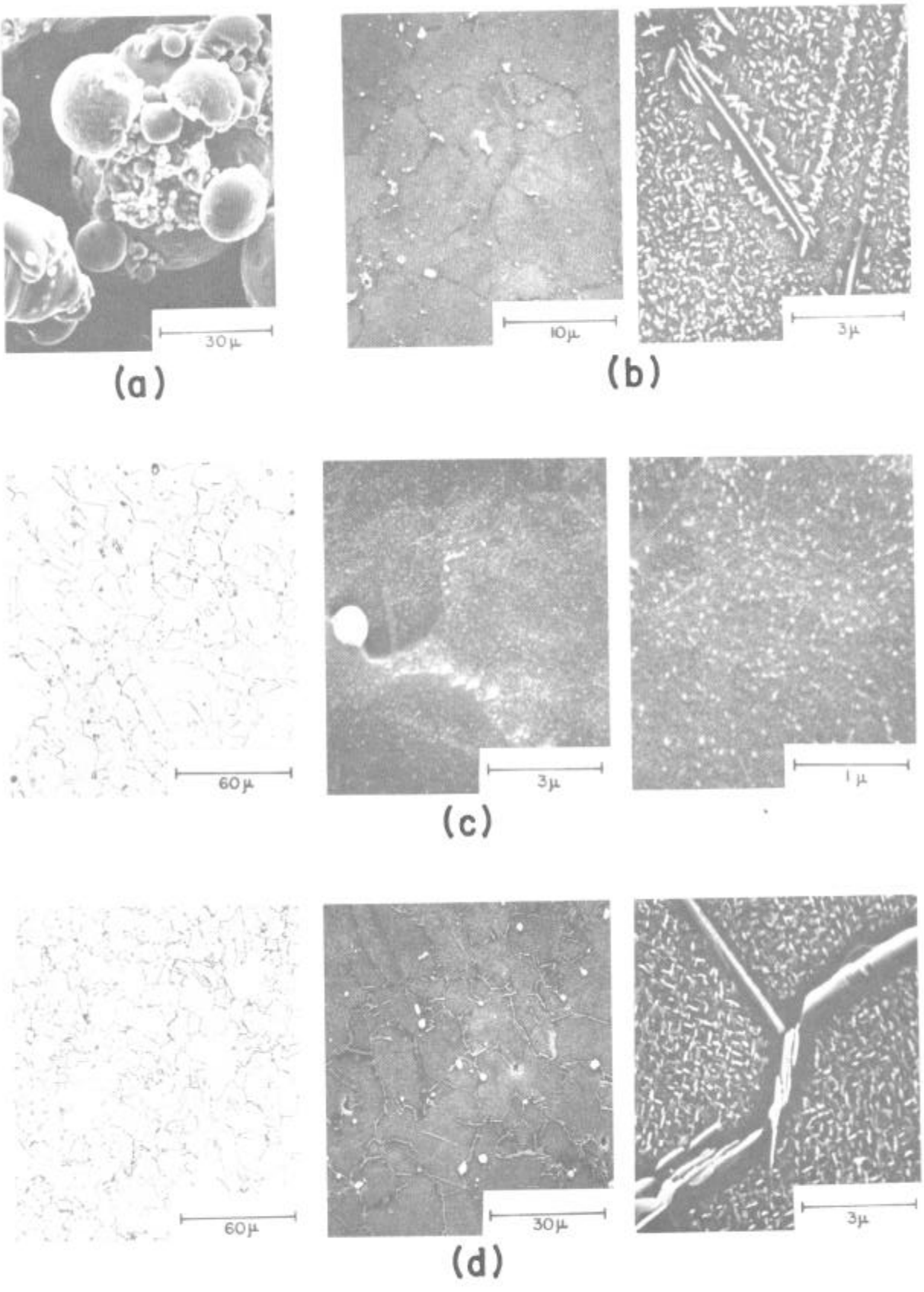
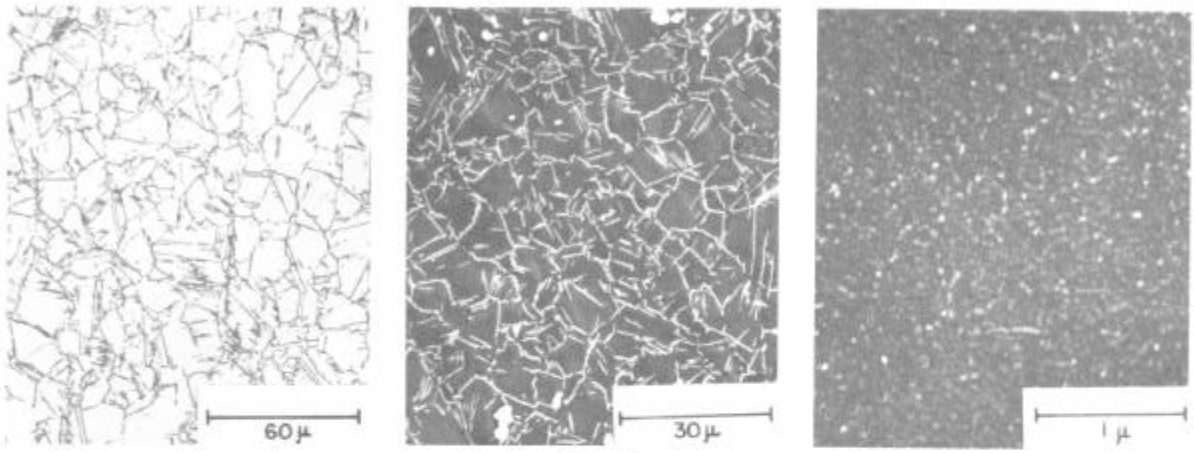
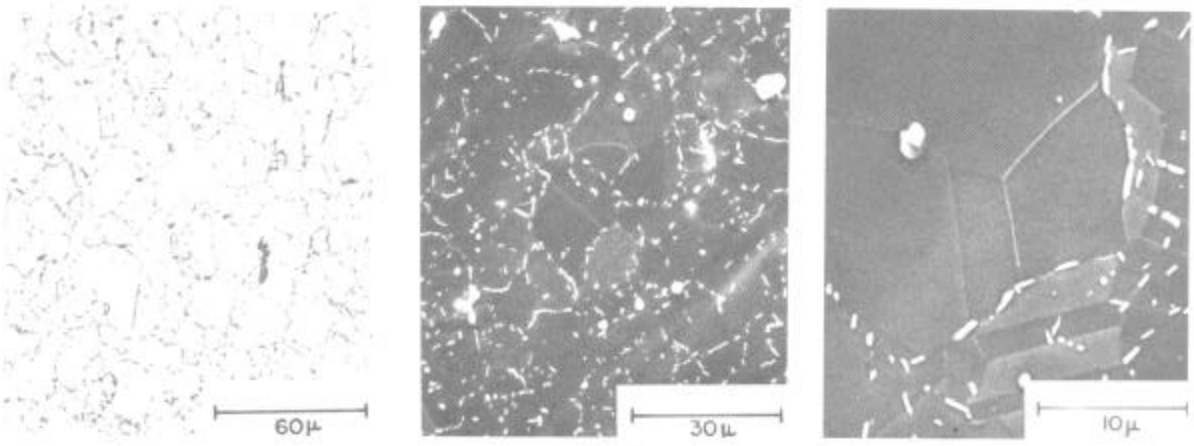


Figure 1: Microstructure of P/M Alloy 718 (a) as-atomized; (b) as-consolidated; (c) as-rolled; (d) as-heat treated, 1600°F/2 hrs/RAC + standard double age





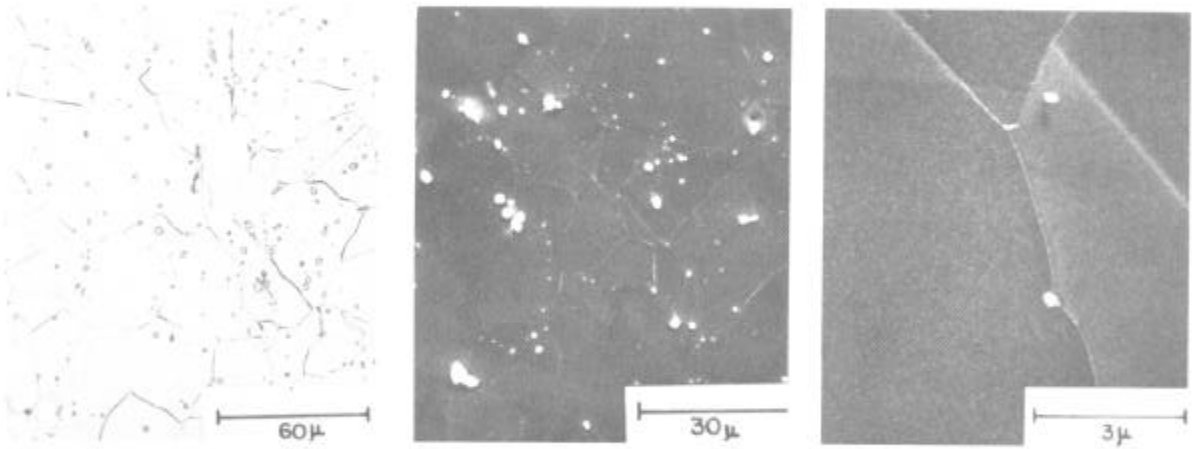


Figure 2: Microstructure of P/M Alloy 718 as-heat treated, (a) 1700°F/2 hrs/RAC + standard double age; (b) 1800°F/2 hrs/RAC + standard double age; (c) 1900°F/2 hrs/RAC + standard double age.

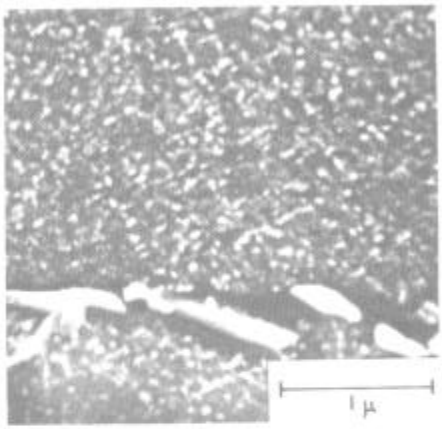


Figure 3: Microstructure of P/M Alloy 718 gradient bar, solution treated for two hours at 1450°F

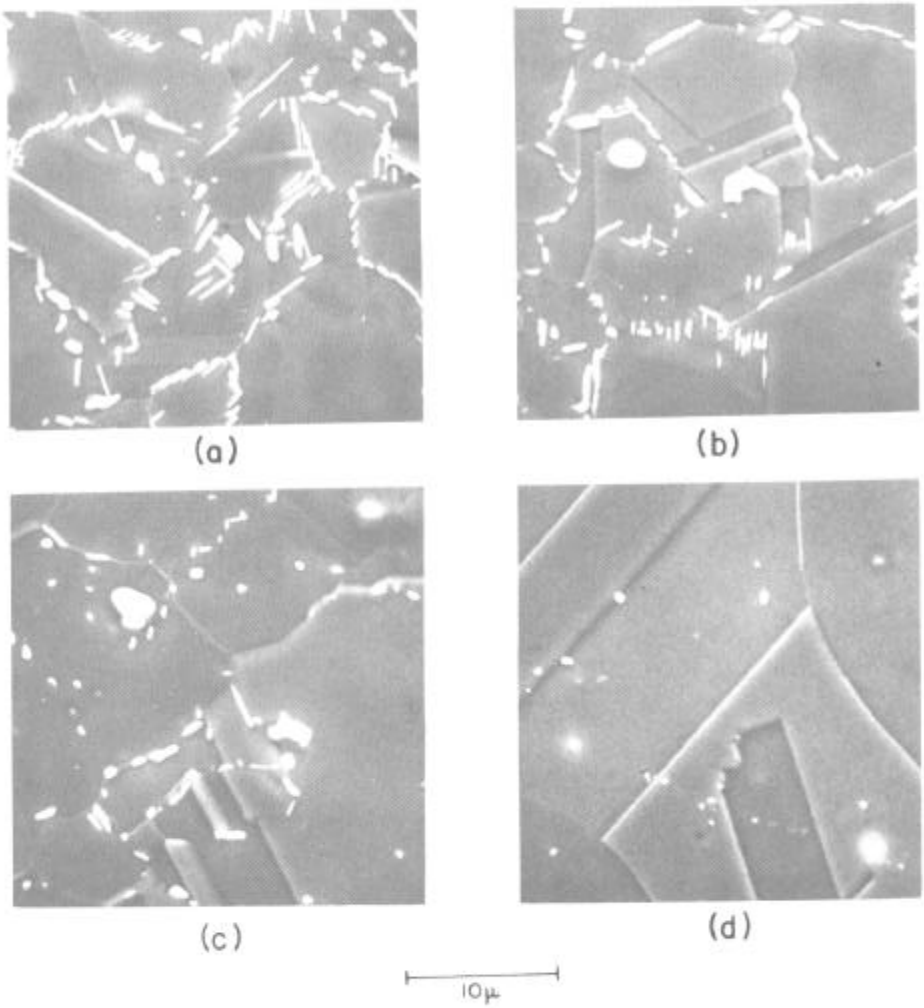


Figure 4: Microstructure of P/M Alloy 718 gradient bar, solution treated for two hours at (a) 1775°F; (b) 1800°F; (c) 1825°F; (d) 1850°F

X-Ray Identification of Phases

The X-ray analyses of extracted residues of powder Alloy 718 revealed the same phases as commonly found in wrought Alloy 718. A strong columbium-rich MC-type carbide and smaller amounts of nitrides and carbonitrides (TiN and TiC, N) were present throughout all the samples. The as-consolidated material and rolled samples solution treated at 871°C (1600°F) and 927°C (1700°F) contained a small amount of Laves phase. Varying amounts of delta were observed between 788°C (1450°F) and 996°C (1825°F), while gamma double prime was evident from 788°C (1450°F) to 871°C (1600°F). The presence of gamma prime was not determined, as it is difficult to detect by X-ray methods in the presence of gamma double prime because of coincidence of the main diffraction peaks.

Mechanical Property Characterization

The tensile strength results presented in Table II and Figures 5(a) and (b) are comparable to those obtained in cast and wrought Alloy 718 bar, and are above the industry specification goals. At both room and elevated temperature, strength levels generally decrease with increasing solution temperature. This behavior follows that demonstrated by cast and wrought Alloy 718 (3,4). Both grain coarsening and increased solutioning of the delta phase contribute to these effects.

Table II. Mechanical Properties of P/M Alloy 718

TENSILE RESULTS

Test Temperature	Heat Treatment	UTS (ksi)	0.2% YS (ksi)	Elongation (%)	Reduction of Area (%)
68°F	AMS5662	185.0	150.0	12.0	15.0
	A	216.7	179.3	17.9	33.5
	A	216.0	185.1	13.1	21.9
	B	212.3	182.4	14.8	26.5
	B	212.4	177.1	15.6	23.6
	C	212.5	173.4	20.7	40.4
	C	210.4	172.9	21.0	42.3
	D	207.0	180.6	22.2	48.4
	D	206.7	180.6	21.7	48.7
1200°F	AMS5662	145.0	125.0	12.0	15.0
	A	172.3	154.6	19.7	32.5
	A	172.1	151.5	15.3	23.9
	B	168.6	150.7	12.1	25.2
	B	171.9	153.3	13.8	28.6
	C	173.0	158.2	9.5	16.7
	C	171.4	147.6	12.1	17.9
	D	163.6	143.9	13.9	17.0
	D	162.5	147.1	9.7	16.7

STRESS-RUPTURE RESULTS: 1200°F, 110 ksi

Heat Treatment	S/R Life (hrs)	Elongation (%)	Reduction of Area (%)
AMS5662	23.0	4.0	-
A	59.1	4.9	13.9
A	48.5	8.9	14.4
B	56.9	8.6	13.3
B	43.4	6.0	14.9
C	61.8	7.7	13.9
C	65.9	7.4	12.3
D	107.4	6.2	13.2
D	115.9	12.1	35.8

Heat Treatments:
A = 1600°F/2 hrs/RAC + 1325°F/8 hrs/furnace cool to 1150°F/8 hrs/AC
B = 1700°F/2 hrs/RAC + 1325°F/8 hrs/furnace cool to 1150°F/8 hrs/AC
C = 1800°F/2 hrs/RAC + 1325°F/8 hrs/furnace cool to 1150°F/8 hrs/AC
D = 1900°F/2 hrs/RAC + 1325°F/8 hrs/furnace cool to 1150°F/8 hrs/AC

Room temperature tensile ductility increases with increasing solution temperature, but the ductility data for the 649°C (1200°F) tensile tests show a downward trend with increasing solution temperature. The largest change in ductility occurs between the 927°C (1700°F) and 982°C (1800°F) solution treatments. Again, grain coarsening and delta phase solutioning were observed in this temperature range. The decrease in tensile ductility at 649°C (1200°F) with increasing solution temperature would indicate that a change in the fracture mechanism is operative at the elevated temperature.

In terms of stress-rupture results at 649°C (1200°F) and 759 MPa (110 ksi) presented in Table II and Figure 5(c), rupture life and ductility are both improved by using a higher solution temperature. The biggest jump in property levels occurs between the 982°C (1800°F) and 1038°C (1900°F) solutioning temperatures. It is in this temperature range that the delta phase completely solutions and grain coarsening takes place more readily. Similar increases in rupture life with higher solution temperatures have been observed in cast and wrought Alloy 718 (5,6). However, the ductility improvement in the P/M Alloy 718 is unique and may involve solutioning of the delta phase without much grain coarsening. Further testing is underway to verify these results.

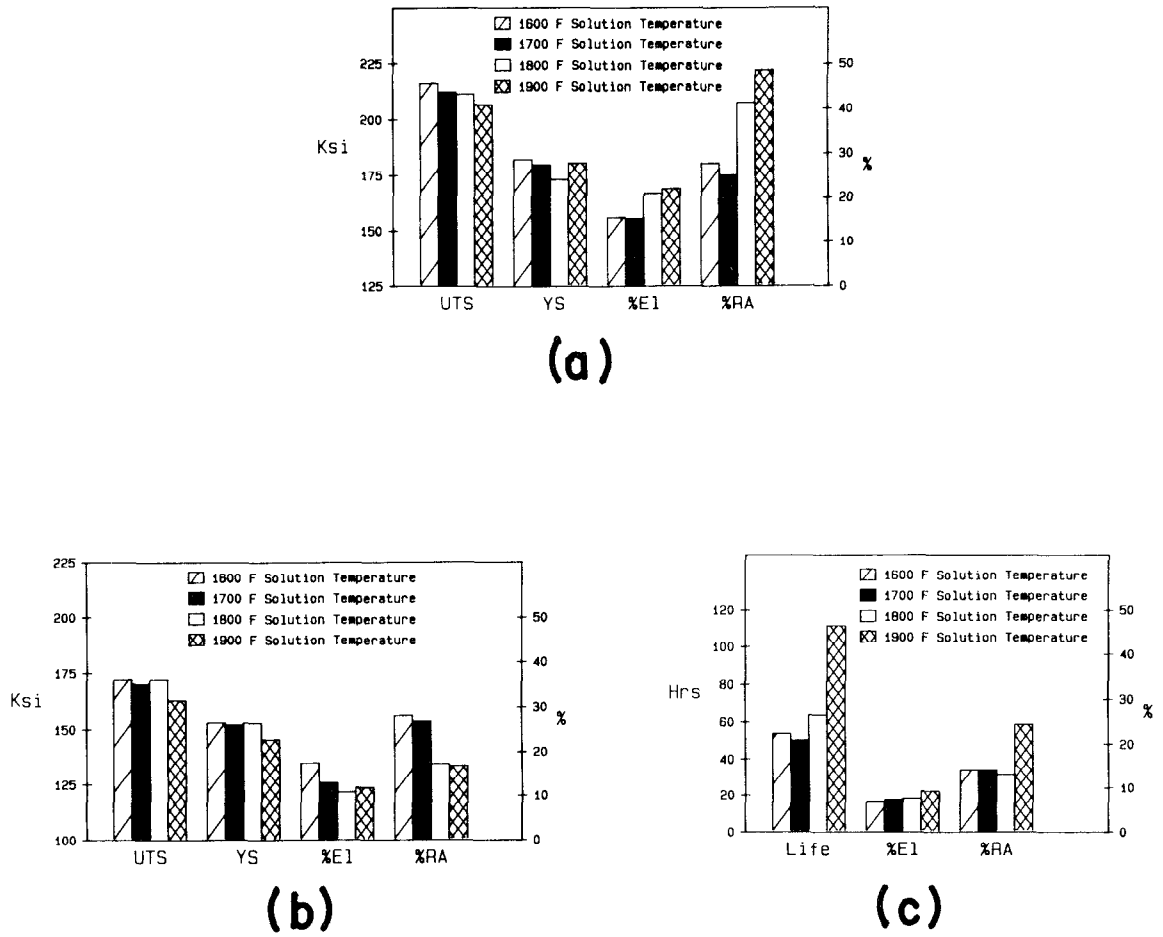


Figure 5: Effect of solution treatment temperature on the mechanical properties of P/M Alloy 718
(a) room temperature tensile properties
(b) 1200°F tensile properties
(c) stress rupture properties at 1200°F, 110 ksi

The occurrence of duplex grains in some of the tested rupture bars was associated with lower rupture life when the bars were solution treated at 871°C (1600°F) and 927°C (1700°F). When such duplex grain bars were solution treated at 982°C (1800°F) or 1038°C (1900°F), the rupture life was unaffected.

The effect of duplex grains on the mechanical properties when solutioned at the lower temperatures may be related to the precipitation of plates of delta phase in the grain boundaries. The presence of larger grains allows the delta to form more continuous plates in the grain boundaries which propagate cracks more readily. The delta phase precipitated during the 982°C (1800°F) solution temperature is more discrete in nature and less in volume. Figures 2 and 4 show the changes in delta precipitation with increasing solution temperature.

Conclusions

The same phases are found in powder Alloy 718 as in its conventionally produced wrought counterpart[7]; however, both precipitate and second phase particles are much finer and more uniformly dispersed in P/M Alloy 718. The observed precipitates appear at approximately the same solution temperatures in the powder alloy microstructure as in the cast and wrought alloy. The recrystallized structure of the P/M material maintains a finer grain size at a given solution temperature, presumably at least partially due to the action of the small discrete particles in pinning grain boundaries. Mechanical properties of P/M Alloy 718 rolled bar are equivalent to those of wrought 718 rolled bar and can be varied as a function of solution treatment temperature. These facts indicate that the use of powder metallurgy technology in production of Alloy 718 provides a high quality product which offers the advantages of a finer and more uniform microstructure and the ability to process at higher temperatures, as well as considerable flexibility in mechanical properties.

References

1) E. E. Brown, R. C. Boettner, and D. L. Ruckle, "Minigrain Processing of Nickel-Base Alloys", pp. L-1-9 in Proceedings of the Second International Conference on Superalloys Processing, Seven Springs, PA, Sept. 1972.

2) W. J. Boesch and H. B. Canada, "Precipitation Reactions and Stability of Ni_3Cb in Inconel 718", Proceedings of the International Symposium on Structural Stability in Superalloys, Vol. 2, Seven Springs, PA, Sept. 1968.

3) Donald R. Muzyka and G. N. Maniar, "Effects of Solution Treating Temperature and Microstructure on the Properties of Hot Rolled 718 Alloy", Metals Engineering Quarterly, November 1969, pp. 23-37.

4) J. D. Buzzanell and F. J. Rizzo, "The Effect of Thermomechanical Variations on the Structure and Properties of Unitemp 718", Universal-Cyclops Internal Technical Report No. R-850, Sept. 1969.

5) H. L. Eiselstein, "Metallurgy of a Columbium-Hardened Nickel-Chromium-Iron Alloy", ASTM STP No. 369, 1965, pp. 62-79.

6) R. C. Hall, "The Metallurgy of Alloy 718", Journal of Basic Engineering, Sept. 1967, pp. 511-516.

7) W. H. Couts and J. F. Radavich, "Factors Affecting Delta Phase Precipitation and Growth at Hot Work Temperatures for Direct Age Inconel 718", paper to be presented at the Fifth International Symposium on Superalloys, Seven Springs, PA, Oct. 1984.