MICROSTRUCTURE AND MECHANICAL PROPERTIES OF INCONEL 625 AND 718 ALLOYS PROCESSED BY POWDER INJECTION MOLDING

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Abstract

Powder injection molding of superalloys offers a potential route for the manufacture of small and geometrically complex components for the aircraft engine industry. In this investigation, 625 and 718 alloy powders with an average size below 10 μm were injection molded into tensile test bars which were then sintered in a hydrogen atmosphere at diverse temperatures after debinding. Other 718 alloy samples were sintered in vacuum. The as-sintered 625 alloy showed an improvement in the mechanical properties over the cast material. The 718 alloy has been difficult to process via PIM because of the presence of a low melting point niobium-rich compound during the sintering process. Differential thermal analysis (DTA), dilatometric, and microstructural analyses were conducted to characterize and optimize the PIM ability of this alloy. In the present work the relationship between PIM, microstructure and mechanical properties of these two alloys is discussed.

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Introduction

In the last decade significant advances have been made in the manufacturing of superalloys via powder metallurgy (P/M) routes. The P/M process overcomes the severe macrosegregation problems that occur during the solidification of high strength nickel-base superalloys ingots. Because of the high purity inert manufacturing environment and the rapid solidification associated with metal powders production, the resulting components usually are of high cleanliness and have a uniform chemistry and fine microstructure. These qualities make P/M powders suitable for use in the manufacturing of critical aircraft engine components by near or net-shape techniques such as isothermal forging and hot isostatic pressing.

Advances in technology have allowed the production of high performance materials like superalloys with high melting points. However, advances in structural design now require components with more complex geometries, which cannot be made by either casting because of the segregation and microstructural inhomogeneity, or by standard P/M processes because of the shape complexity and dimensional tolerances. In addition to the high cost associated with these manufacturing processes, a high volume production of small parts using these processes makes it prohibited. Therefore, powder injection molding (PIM) represents an alternative manufacturing process for the production of these complex components. Unfortunately, very little work has been published and very little effort has been directed to the development of superalloy PIM technology [1]. Therefore, in an effort to develop this technology the National Center for Excellence in Metalworking Technology under the Mantech Program is conducting work in this field.

In this investigation, the effects of sintering variables on the microstructure and mechanical properties of Inconel 718 and 625 produced by PIM processing are examined.

Experimental

Tensile bars and cylindrical specimens of Inconel 718 and 625 alloys were produced by powder injection molding from powders produced by Ultrafine Powder Technology. Both powder materials had an average particle size of 8.5μm with typical compositions shown in Table I.

Table I Chemical Compositions of 625 and 718 Alloy Powders

Alloy	Ni	Cr	Nb	Mo	Al	Ti	Fe	Si	Mn	C	S	O	N
625	62.23	22.3	3.8	8.8	.034	.011	3.4	.36	.012	.010	.002	.032	.004
718	50.70	19.6	5.21	3.21	.59	.98	19.4	.13	.10	.042	.007	.038	.023

Alloy 625

The prealloyed powder was mixed with a wax/polymer binder mixture to a 66% solids loading, then injection molded into 1.25 cm diameter and 1.25 cm length cylindrical specimens, and flat ASTM-type tensile specimens [2]. The specimens were initially debound by solvent immersion in heptane; subsequent thermal debinding was performed under a hydrogen atmosphere for one hour in a retort furnace at ~550°C. Immediately after debinding, the samples were sintered in a hydrogen atmosphere in a temperature range of 1288 to 1298°C for 24 to 60 minutes. During sintering, the samples were stepped along a horizontal furnace at predetermined distances and held for specified times. The purpose of this procedure was to avoid excessive thermal gradients and to assure a homogeneous sintering. After sintering, the samples were allowed to cool away from the hot zone.

The Archimedes method was used to determine the density of the specimens in the as-sintered condition. Other specimens were prepared for metallographic analysis.

The PIM tensile specimens after sintering had a gauge section width of approximately 5.6 mm and thickness of 2.75 mm. Prior to mechanical testing, the specimens were tumbled in an

abrasive media to remove surface imperfections. Testing was performed in accordance with ASTM E-8 standard.

Inconel 718

Two sets of flat tensile specimens were prepared by PIM using two different binder mixtures. The first set was prepared in a similar manner as the 625 alloy and the second by using a proprietary binder mixture from Parmatech [3].

Dilatometric and differential thermal analyses (DTA) were conducted to understand the possible phase changes which might adversely affect the sintering behavior. The DTA was done on pressed powder in an argon atmosphere at temperatures up to 1350°C using 10°C/min heating and cooling rates. Dilatometric studies conducted in a hydrogen atmosphere were also performed to understand the sintering behavior of the material. This was done on cylindrical plugs pressed to a density comparable to injection molded Inconel compacts, at temperatures up to 1260°C for 15 hours with an intermediate hold at 1150°C for 2 hours and a 10°C/min heating and cooling rate.

Sintering was conducted using both hydrogen and vacuum environments. The sintering in hydrogen was performed at 1260°C for 6 hours with an intermediate hold at 1150°C for two hours and a 10°C/min heating and cooling rate. The vacuum sintering was carried out at a pressure of approximately 10^{-3} to 10^{-4} torr. A partial pressure of argon was backfilled into the furnace to prevent chromium evaporating from the alloy. The sintering temperatures were 1250, 1260 and 1275°C, with holding times varying from 1 to 8 hours and ramp rates from 1 to 25 °C/min. In some trials, an intermediate hold at 1150°C for 2 hours was used.

Vacuum sintered samples were solution heat treated at 950 °C for 1 hr in an inert environment and air cooled. The precipitation heat treatment was 718 °C for 8 hours, furnace cool at 38 °C per hour to 620 °C, hold for 8 hours and then air cool [4]. One set of samples was hot isostatically pressed (HIP) at 1190 °C for 4 hours at 15 ksi (103.5 MPa).

Density measurements were conducted on the as-sintered and hipped samples. Tensile testing was performed on the as-sintered and heat treated samples in accordance with the ASTM E-8 standard. Further, metallography was performed on selected samples.

Results and Discussion

Table II shows the sintering processing parameters, the average densities attained, and the resulting average mechanical properties of at least two specimens of the 625 alloy. The results indicate that densities are above 99% of that of wrought material (8.44 g/cm^3) [5]. This is expected since the temperatures used for sintering were at or slightly above the solidus for as-cast and wrought Alloy 625 (~1288 and ~1290 °C respectively) [5], Using these high temperatures, long holding times were not required to achieve high densities. To achieve high densities in this material was not a problem. However, the resulting microstructure was adversely affected (non-uniform grain size) by the development of thermal gradients in the

Table II. Density and Tensile Properties for Alloy 625 at Different Sintering Schedules

Run No	Temp ℃	Hold time min	Density %	YS 0.2% Mpa	UTS MPa	% Elong	Travel interm min	Travel incr. cm	Grain size* µm
1	1298	60	99.7	347.3	665.5	36.9	00	0.0	>175
2	1288	60	99,9	342.3	687.3	95.3	15	5.1	85
3	1288	60	100	344.3	805.8	48.0	10	10.2	85 (42)
4	1289	30	99.3	400.6	751.9	80.7	5	10.2	46
5	1288	24	99.8	376.2	848	46.7	4	10.2	20 (19)

( ) Grain size measured in the transverse direction.

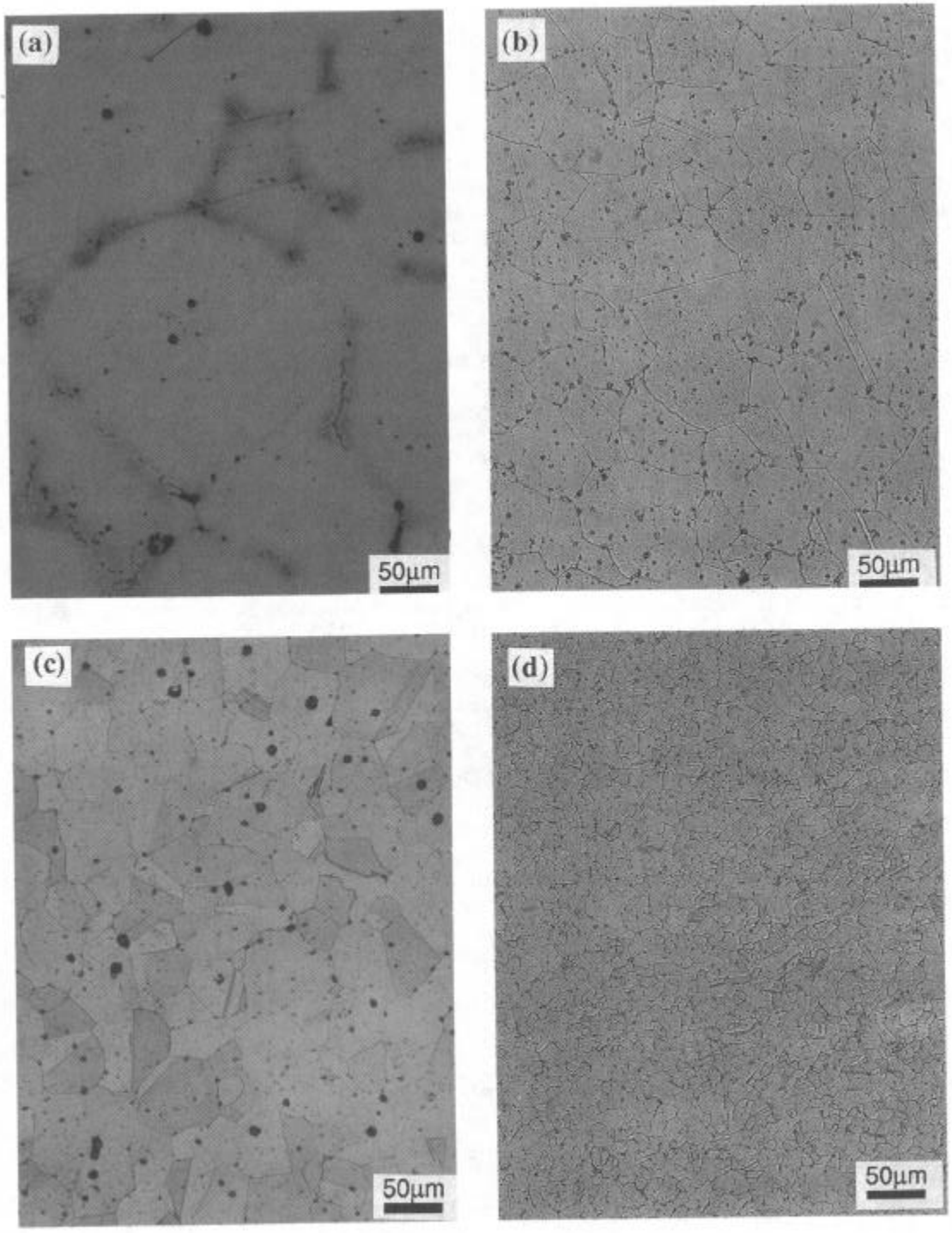

Figure 1 - Optical microstructures of PIM Inconel 625 after sintering at; (a) 1298°C/60 min., (b) 1288°C/60 min., (c) 1289°C/30 min. and (d) 1288 °C/24 min.

horizontal pusher type furnace. Thus, in order to minimize the thermal gradient, the samples were moved at given length increments for specified times. This incremental travel of the specimens demonstrated that the thermal gradients had a pronounced effect on the microstructure, which subsequently had an effect on the mechanical properties as evidenced by the scatter shown in Table II.

Figure 1 shows some of the microstructures obtained from Runs 1, 3, 4 and 5. In Run 1 (Figure 1a) the specimens were not pushed through the furnace thus, this resulted in an excessive and irregular grain growth (>175μm). In this case, however the lower mechanical properties achieved are due primarily to incipient melting at the grain boundaries caused by the high sintering temperature, as well as by precipitation of Laves phases at the grain boundaries.

A full densification was achieved at 1288°C using an incremental travel of 10.2 cm every 10 minutes and a total sintering time of 60 minutes (Run 3). However, the grains were found to have an irregular size (85μm transverse and 42 μm longitudinal) with a maximum aspect ratio of ~2:1. Presumably, this is due to the temperature gradient of the furnace.

In contrast, shorter holding times resulted in a more uniform grain size. For instance, after a total holding time of 24 min at 1288°C a grain size of 20μm in longitudinal and 19μm in the transverse direction of the samples was obtained. Figures 1c and 1d show the microstructures for 30 and 24 minutes holding times respectively. The results suggest that shorter total holding times during incremental travel produce a more uniform and smaller grain size. This translates into a relatively higher yield strengths than the specimens sintered for longer periods of time. The comparatively low UTS values measured in Run 4 are presumably due to the lower densities observed.

In general, mechanical properties from Inconel 625 produced by PIM are comparable to or better than those of the cast and solution treated material. Additionally, the observed PIM microstructures also indicate that substantial improvements can be made by additional heat treatment. This opens a new avenue for the manufacture of components from this material with complex geometries.

Sintering of PIM Inconel 718 is much more difficult than that of the 625 alloy. Some of the problems encountered can be related to the solidification behavior of this material. For example, it has been observed that a Nb-bearing γ/Laves eutectic phase can be produced at ~1180°C during solidification [6,7]. Laves phases can also be formed during annealing at ~1038 °C [8]. Although, the rapid solidification of the atomized powder produces a relative uniform microstructure. However, the high temperatures associated with sintering of the PIM material (close to the melting point) and the complex precipitation reactions which occur in the solid state during heating, can lead to the formation of a γ/Laves-eutectic phase.

The DTA thermogram "on-heating" shown in Figure 2a shows a slight depression at approximately 1170°C. This indicates the formation of the γ/Laves eutectic phase and correlates well with other studies [6, 7, 9]. Also, the minimum on the curve at 1348°C indicates the liquidus for this material. In the "on-cooling" portion of the thermogram, the liquidus was observed at a lower temperature (~1333°C) due to the supercooling effect of the alumina crucible. Although, the solidus was not evident from the DTA curves, it is suspected to occur at approximately 1300 °C on cooling, and possibly is slightly higher on heating.

Figure 2b shows the dilatometric behavior of pressed Inconel 718 powder. A volume change due to the γ/Laves eutectic phase at 1180°C was not observed. However, immediately after the maximum temperature was achieved (1260°C), a sudden shrinkage was observed to occur in less than one hour. This indicates that sintering of this material can be achieved in a relative short time. However, density measurements of the specimens (~86% fractional density) indicate that a complete densification was not obtained even after ~17 hours at 1260°C. Metallographic analysis showed that the specimens had a densified outer layer with a small porous core. Since full density was not observed, it is reasonable to assume that the hydrogen environment has played a role in inducing porosity in Inconel 718.

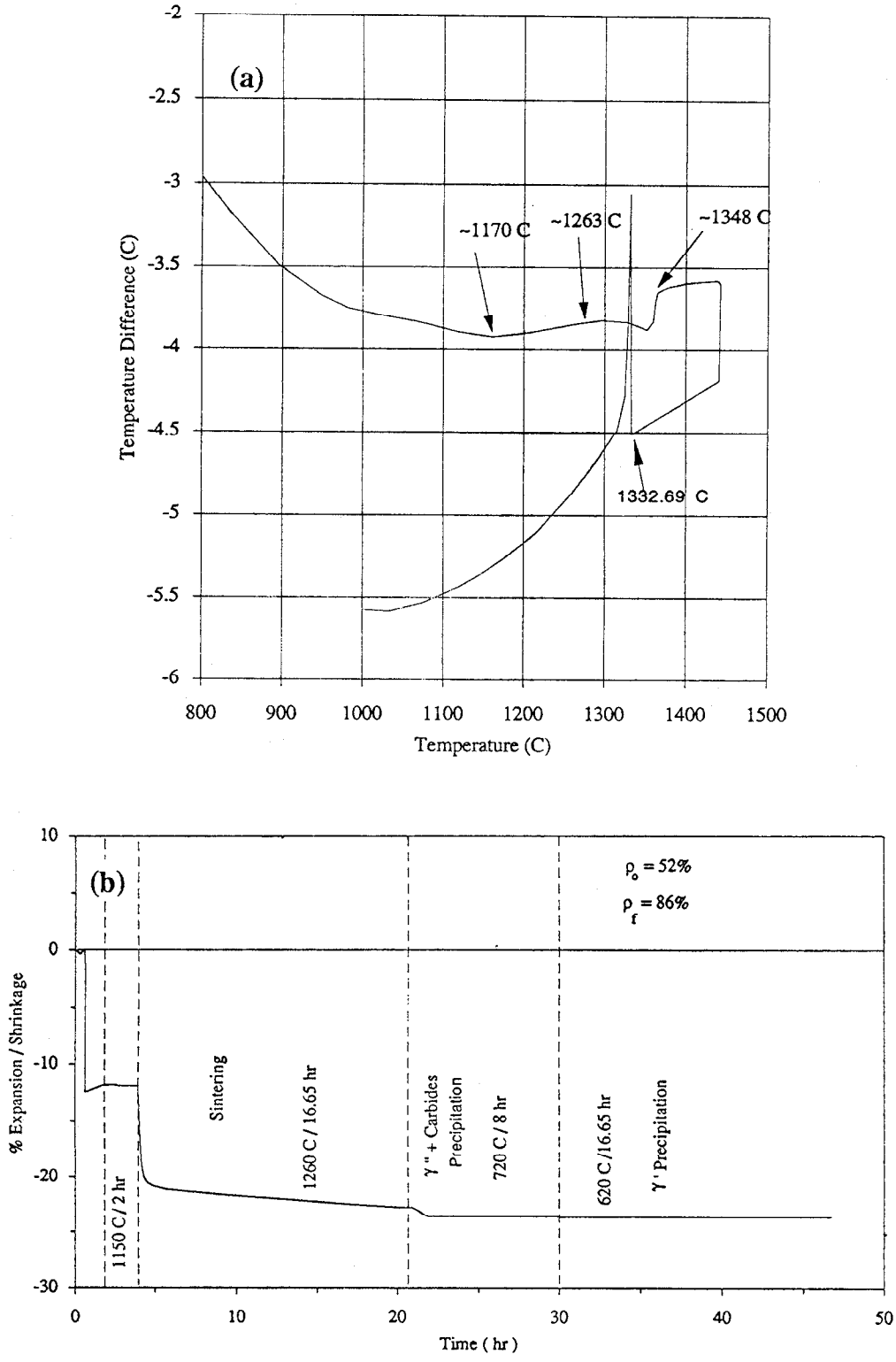

Figure 2 - (a) DTA thermogram and (b) Dilatometry thermal cycle for Ultrafine Inconel 718 powder.

SEM-EDS analyses of specimens sintered in hydrogen also suggest that Laves phases can be formed. Figure 3 shows the BEI microstructures obtained from a specimen containing both partially and fully sintered regions. Note that, in both cases the powders and the grain boundaries are surrounded by a Nb-containing phase. EDS analysis indicates that they have a Laves phase origin, since their composition {44Ni-16.4Cr-16Fe-12.5Nb-3.2Mo-1.4Si-Ti-Al} is consistent with that of Laves phases [6]. This composition persisted even after diffusion had occurred during the sintering cycle. A proportion of approximately 5-7% was found within the microstructure.

Interestingly, sintering in hydrogen also produced a peripheral ring of dense material surrounding a porous core in a similar manner as observed in the dilatometric specimens. This is expected since, as the densification of the specimen progresses in from the surface, hydrogen remains (or water vapor formed by the reaction of hydrogen with the oxygen in the oxide film on the powders) entrapped at the center and has difficulty diffusing out, resulting in porosity. Therefore, the sintering of Inconel 718 in a hydrogen environment may be impossible.

Table III. Sintering Schedules and Densities Obtained before and after Hipping

Run No	Temp. ℃	Ramp Rate °C/min	Interm. Hold(*) Hrs	Holding Time Hrs	Density Sintered (g/cm^3)	Density HIP'd (g/cm^3)
1	1250	1	0	2	7.81	7.90
2	1250	1	2	8	7.96	7.78
3	1250	8	2	2	7.55	7.44
4	1250	8	0	8	8.12	8.17
5	1275	1	2	2	7.71	7.87
6	1275	1	0	8	8.18	8.21
7	1275	8	0	2	7.90	8.11
8	1275	8	2	8	7.94	8.16
9	1262	4.5	1	5	7.76	7.94
10	1250	10	2	1	7.70	NA(**)
11	1260	20	0	2	7.95	NA(**)

(*) Intermediate hold at 1150°C. Ramp rate 5°C/min from RT to 600°C. From 600°C to intermediate or final temperature at the specified ramp rate.
(**) Not HIPed.

Vacuum sintering of PIM Inconel 718 was conducted using the schedules indicated in Table III. This shows both the effect of the sintering schedule and hipping on the density. Note that HIP has a relative minor effect in increasing the density. (The low density values for Runs 2 and 3 after hipping are within the statistical scatter error), Instead, it appears that longer final holding times during sintering have the largest effect. Note that, in general, the specimens sintered with an intermediate hold at 1150°C for 2 hours have low density values. This might be expected since there is the possibility of incipient melting of Laves phases at this temperature. The fraction of liquid (with the Laves phases composition) will increase with temperature and it may remain at the final sintering temperature: thus, during cooling it may produce porosity due to shrinkage (Figure 4).

The effect of the ramp rate is not evident from the above table. However, by plotting the density after sintering versus the ramp rate for those trials with a short holding time (Figure 5), it appears that density increases with the ramp rate. The explanation for this effect is not clear; however, it is reasonable to assume that higher heating rates may decrease or prevent the

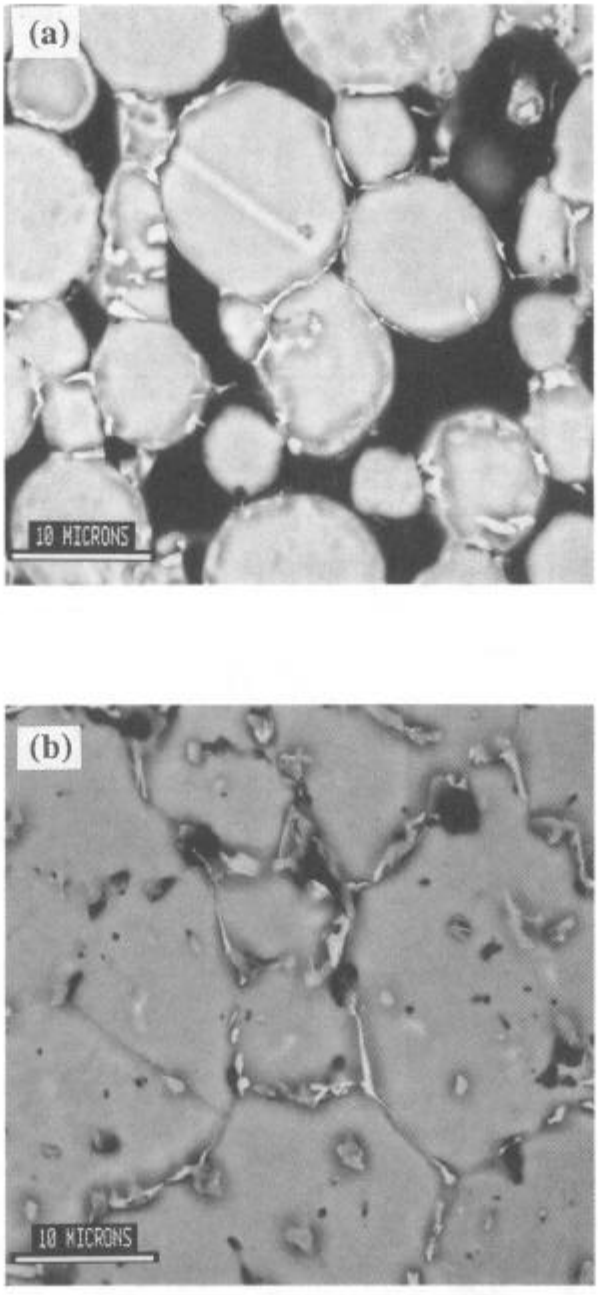

Figure 3 - Inconel 718 sintered in hydrogen at 1260°C for 6 hours after an intermediate holding at 1150°C for 2 hours. A 10°C/min. heating and cooling rate was used; (a) partially sintered region at center and (b) denser region at the periphery.

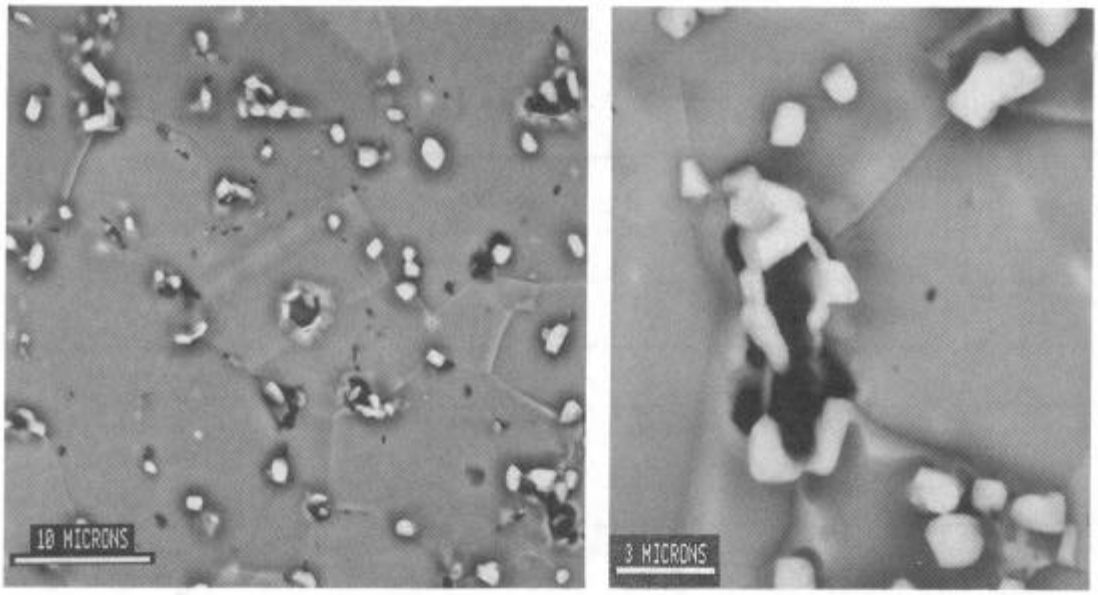

Figure 4 - BEI microstructures of PIM Inconel 718 sintered in vacuum at 1275°C/2 hours, with 2 hour holding at 1150°C.

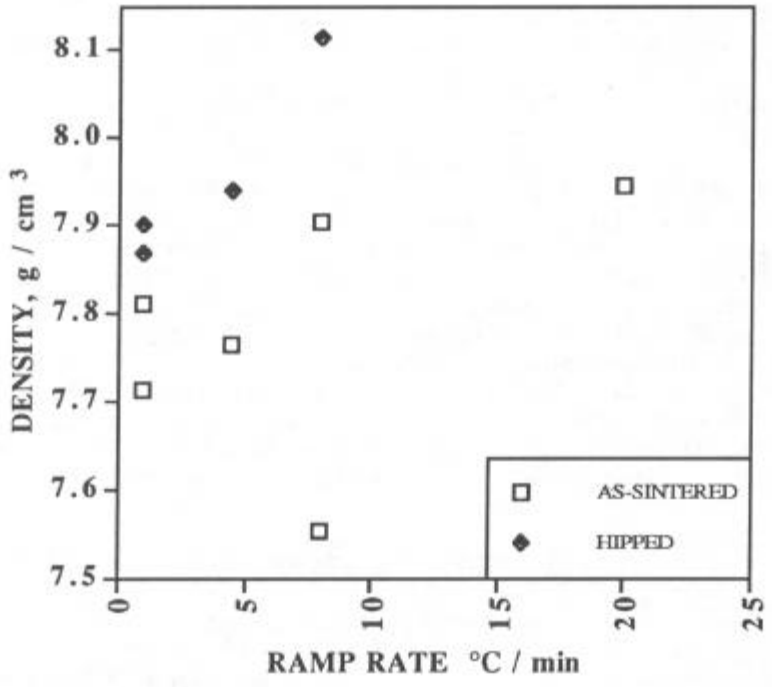

Figure 5 - Effect of the ramp rate during sintering of PIM Inconel 718 at 1250 °C, 1260°C and 1275°C with holding times up to 5 hours.

formation of heavy intermetallic eutectic Laves phases which may cause porosity during cooling (note that the starting material is rapidly solidified powder with practically no segregation which can lead to the formation of Laves-phases). Work is in progress to clarify this effect.

Table IV shows selected tensile mechanical properties of as-sintered and precipitation heat treated material, and are compared with those specified in AMS 5662 G [4].

Table IV. Tensile Properties of PIM Inconel 718 after Precipitation Heat Treatment

Sample #	Interm. Holding t at 1150°C hrs	Sintering T (Hold t) °C/(hrs)	Ramp Rate °C/min	Density g/cm^3 Sinter/HIP	0.2% YS MPa (ksi)	UTS MPa (ksi)	% Elong
Inconel 718(1)		BASE	LINE		1036 (150.0)	1278 (185.0)	12.0
2	2	1250 / (8)	1	7.96 / 7.78	794 (115.0)	916 (132.6)	5.0
4	0	1250 / (2)	8	8.12 / 8.17	963 (139.4)	1218 (176.4)	11.3
5	2	1275 / (2)	1	7.71 / 787	686 (99.4)	812 (117.5)	5.0
6	0	1275 / (8)	1	8.18 / 8.21	972 (140.7)	1199 (176.3)	17.4
7	0	1275 / (2)	8	7.90 / 8.11	995 (144.1)	1187 (171.9)	6.9
8	2	1275 / (8)	8	7.94 / 8.16	818 (118.5)	892 (129.1)	3.3
12(2)	0	1260 / (2)	20	7.36 / N.H	931 (134.8)	1191 (172.5)	7.0

(1) Inconel 718 base line mechanical properties after precipitation heat treatment as per AMS 5662G [4].

(2) Mechanical properties measured in the as-sintered condition.

This table indicates that the best combination of YS, UTS and ductility is obtained by sintering at 1275°C for 8 hours after a ramp rate of 1°C / min. (Sample 6). In contrast, the specimens with an intermediate hold at 1150°C for 2 hours have the lowest properties. This is presumably due to the low density caused by the large proportion of Laves phases formed at the intermediate hold. The relatively low mechanical properties also suggest that these phases were not completely dissolved in the γ-matrix during sintering at high temperature or during hipping and heat treatment. Interestingly, Sample 12, sintered at 1260°C for 2 hours at a ramp rate of 20°C/min without an intermediate hold and precipitation heat treatment, showed at least 90% of the yield strength and UTS of the base line material [4]. The low ductility observed in Sample 12 may be attributed to the low density. This is be expected since sintering was done for only 2 hours and at a lower temperature than Sample 6. Nevertheless, the ductility of this material is higher than the precipitation heat treated specimens with an intermediate hold and lower heating rates.

Summary and Conclusions

The results suggest that powder injection molding of Alloy 625 is possible. Densities above 99% of the theoretical value can be achieved without producing undesirable microstructures. Material with a uniform and fine grain size can be obtained by sintering in hydrogen for a short period of time at temperatures close to the melting point. Also, the tensile properties of the as-sintered material are similar to or better than those for cast and heat treated material.

Sintering of PIM Inconel 718 appears to be more complicated because of the formation of eutectic-Laves phases. Although, these phases were not completely evident in the DTA data, EDS analysis indicated their presence even after long times at the sintering temperatures. In

contrast to Alloy 625, sintering of Inconel 718 in a hydrogen environment results in a poor density, Densities in the range of 90 to 99% of the theoretical were achieved when sintering in vacuum. The higher densities were obtained using higher temperatures and long holding times. Also higher ramp rates during heating appeared to have a positive effect on the density, whereas hipping at 1190 °C for 4 hours at 103.5 MPa (15 ksi) had a minor effect on the density. The best combination of tensile properties and ductility were observed in specimens which were not subjected to an intermediate holding during sintering. Also, these specimens achieved at least 90 % of the base line yield strength. These results are very encouraging and suggest that substantial improvements can be made by further microstructural control of the PIM material.

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