Metal Injection Moulding of Heat Treated Alloy 718 Master Alloy

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Master alloy powders provide a cost-effective alternative to pre-alloyed powders and enhance the properties of the blended Metal Injection Moulding (MIM) feedstock. The mechanical properties and microstructural evaluation of injection moulded and heat-treated Alloy 718 master alloy powder, developed specifically for MIM, are presented. The investigation is a continuation of earlier work, which determined that a homogenous microstructure is produced on sintering and the tensile properties are comparable to cast and annealed material. The microstructural evaluation by optical microscopy revealed a homogenous distribution of precipitates. Fracture surfaces were investigated by scanning electron microscopy and the species of precipitates highlighted on the fracture surface of the sintered material was identified by Energy Dispersive X-ray (EDX) analysis. The results of the mechanical tests at 20°C and 540°C are compared with both wrought properties and MIM components produced using pre-alloyed Alloy 718.

1. Introduction

The emerging interest in Metal Injection Moulding (MIM) of nickel based super alloys for high temperature applications is highlighted by the large-scale production of MIM automotive turbocharger components in Europe and the US. MIM turbocharger components are currently limited to applications with working temperatures less than 500°C. They are typically manufactured using heat-resistant nickel based alloys and stainless steels, in some cases via a master alloy route. The most demanding high temperature applications however require nickel-based super alloys, including Inconel 718. Alloy 718 is a γ'' precipitation-hardenable nickel-chromium alloy containing significant amounts of iron, niobium, and molybdenum along with lesser amounts of aluminium and titanium. The alloy combines corrosion resistance and high strength with outstanding weldability, including resistance to postweld cracking, due to the low carbon level. The alloy has excellent creep-rupture strength at temperatures to 1300°F (700°C) and is commonly used in aerospace engines and stationary gas turbines, rocket motors, spacecraft, nuclear reactors, pumps, and tooling. The production of net shaped components via MIM would provide an economical alternative to investment casting and machining.

Master alloy powders have been successfully used in MIM to provide an alternative to pre-alloyed powders for stainless steels, including 420, 316L and 17-4PH as well as tool steel and low alloy steels. Recent results by Davies *et al.* [1] indicated that 316L master alloy powder, blended with carbonyl iron produced sintered 316L components, with tensile properties that exceeded both the MPIF Standard 35 and, significantly, wrought 316L annealed forgings, plate and hot-finished anneal bar stock. The master alloy feedstock exhibited fewer tendencies for powder binder separation near the gate, possessed better green strength and less distortion during debinding. The components can also be pre-sintered at a lower temperature than pre-alloyed powder.

The development of nickel-based superalloy master-alloy powder for MIM was proposed by Davies *et al.* [2] to provide increased resistance to distortion during and prior to debinding. The preliminary results indicated that master alloys can be used in the MIM fabrication of nickel based super alloys to produce high-density sintered parts. The microstructures were comparable to those obtained by MIM using pre-alloyed powder and cast materials, prior to final heat treatment. The results of the Energy Dispersive X-ray (EDX) analysis suggest that the carbonyl Fe and carbonyl Ni powder were successfully diffused into the matrix during sintering, producing a homogenous microstructure. The tensile properties of the sintered material are comparable the properties of both cast and annealed Alloy 718. As expected the as-sintered properties do not match the properties of heat-treated wrought Alloy 718.

Wohlfromm *et al.* [3] provided a summary of results from a comprehensive MIM trial on the following nickel based super alloys; Hastelloy X, Nimonic 90, Inconel 713C, Inconel 100, GMR-235 and Inconel 718. The pre-alloyed powders were moulded using BASF's acetal based binder and sintered in a molybdenum retort furnace, with a 5 K/min heating rate and a hold time of 3 hours at the final sintering temperature. Specifically, Alloy 718 was sintered in an argon atmosphere at 1300°C, which achieved a 97% theoretical density, by liquid phase sintering. The as-sintered material was Hot Isostatically Pressed (HIP) to almost full density and then heat-treated, producing mechanical properties superior to cast alloys and approaching the values of wrought materials, in all cases.

The aim of the present work is to compare the results of the tensile tests of the heat-treated Alloy 718 MIM material produced by the master alloy route with both the properties of wrought material and MIM Alloy 718 produced using pre-alloyed powders.

2. Experimental Procedure

The Alloy 718 master alloy was designed to include additions of both carbonyl Fe and carbonyl Ni with a blending ratio 4:1:1 (Master Alloy: Fe: Ni) to produce the Alloy 718 composition after sintering. The composition specification of the master alloy and pre-alloyed Alloy 718 are both shown in Table 1. The master alloy was melted in an induction furnace within an argon atmosphere and atomised with high-pressure argon gas, using proprietary atomiser design technology, in order to produce fine powder suitable for MIM. The as-atomised powder was air classified to a particle size of 90% minimum -22μm.

The blended powders were mixed with a proprietary binder system, and pelletised for injection molding. The form of the tensile test specimen is important because the MIM tensile specimen must be compared directly with the standards for wrought and sheet material. Tensile test bars, as shown in fig. 1, were produced by injection molding in a single cavity mold designed to comply with EPMA guidelines. The ratio of length/diameter of the tensile test bars was 20/1.

The basic sinter cycle involved a pre-sinter stage ramping to 600°C in hydrogen (7°C/min) followed by a dwell at temperature for 120 minutes, which assisted in the removal of the surface oxides and the residual carbon from the binder and the carbonyl powders. The furnace was then evacuated and the temperature raised by 7°C/min to 1000°C, followed by a decreasing ramp rate stages to the final sintering temperature of 1265°C for 1 hour. The samples were then furnace cooled and back-filled with nitrogen when the temperature dropped below 600°C. Preliminary sintering trials indicated that sintering at 1260°C and 1280°C for longer periods, up to 4.5 hours, produced inferior sinter densities and poor mechanical properties, with as-sintered ultimate tensile strengths of less than 900 MPa.

	Alloy 718 Pre-Alloyed Material			**Alloy 718 Master Alloy Powder**		
	Nominal	Minimum	Maximum	Nominal	Minimum	Maximum
Ni	53.0	50.0	55.0	52.0	50.0	53.0
Cr	19.0	17.0	21.0	28.0	25.5	29.0
Fe	Balance			Balance		
Nb	5.1	4.7	5.5	7.5	7.12	8.25
Mo	3.0	2.8	3.3	4.7	4.20	4.95
Ti	0.9	0.65	1.15	1.4	0.975	1.725
C	0.04	0.03	0.08	0.08	0.045	0.12
Al	0.5	0.2	0.8	0.75	0.3	1.2

Other elements include (%wt. max.); 1 Co, 0.3 Cu, 0.35 Si, 0.35 Mn, 0.015 S, 0.015 P, 0.006 B

Table 1: Alloy IN718 composition specifications (%wt.).

Figure 1: Sintered IN718 tensile test bar.

A number of sintered specimens were heat treated by solution annealing in vacuum at 980ºC for 1 hour and aged at 720ºC for 8 hours and then at 620ºC for an additional 8 hours. The mechanical properties of the sintered and heat-treated specimens were evaluated by tensile testing at room temperature and also at an elevated temperature of 540ºC. The gauge lengths of the specimens were not machined or polished prior to tensile testing. Specimens were prepared of optical microscopy by standard metallographic techniques. The polished surfaces were electrolytically etched (4.5 volts for 10-20 seconds) in a 5% aqueous sulphuric acid solution, which preferentially etches the precipitates. The fracture surfaces were examined by scanning electron microscopy, incorporating EDX analysis to identify the second phase precipitates present.

2. Results and Discussion

The optical micrographs of a polished and etched section of an Alloy 718 as-sintered tensile specimen (fig. 2) and the sintered and heat treated specimen (fig. 3) clearly show a relatively fine grain size less than 15 microns and precipitates, which are characteristic of Alloy 718. The heat treatment has significantly improved the homogeneity of the microstructure, specifically the grain size is reduced and the second phase precipitates are more evenly distributed.

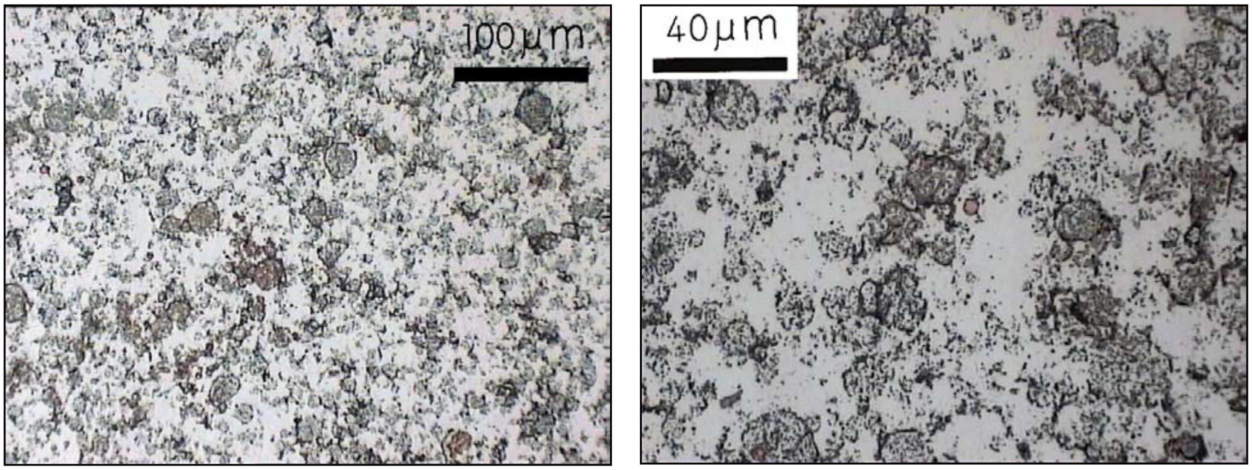

Figure 2: Optical micrographs of Alloy 718 in the as-sintered condition.

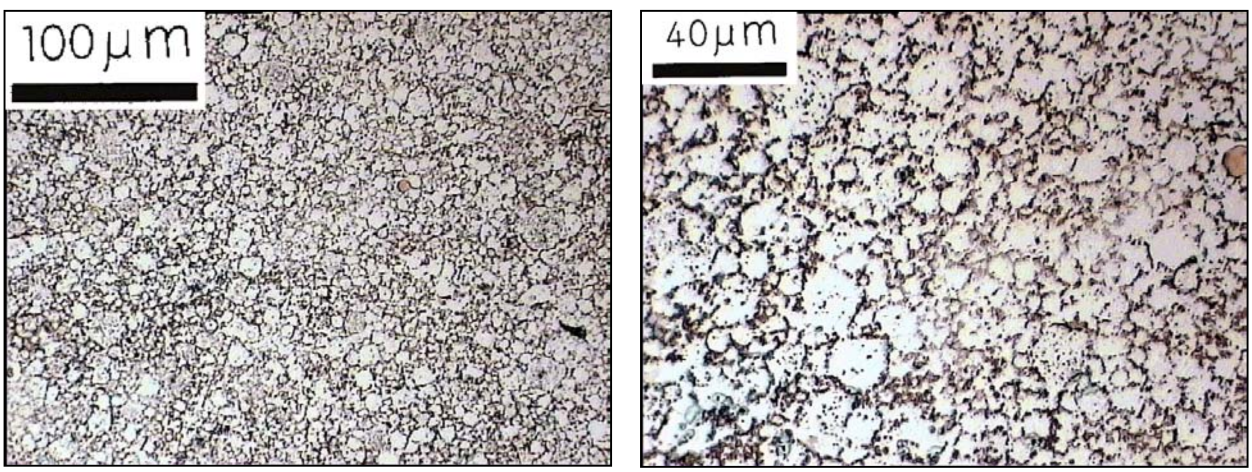

Figure 3: Optical micrographs of Alloy 718 in the sintered and heat-treated condition.

The results of the mechanical tests are provided in Table 2 for MIM Alloy 718 in both the as-sintered and sintered/heat-treated conditions. The results indicate that the heat treatment significantly improved the properties of MIM Alloy 718, especially at the elevated temperature of 540°C. The mechanical properties compare favourably with cast Alloy 718 but fall short of the wrought values, especially in terms of ductility. However, minor surface flaws and the sintered surface texture may have constrained the elongation of the specimens. The fracture surface of the heat-treated material (fig. 4) features a generally ductile fracture surface with a few large prior particles present, typically less than four per specimen, which may restrict ductility of the material. Incorporating a sieving stage before air classifying, to perhaps the finer grade of 90% -16 microns, will probably reduce the incidence of prior particles. Interestingly, relatively few particles were observed in the fracture surfaces of the elevated temperature tensile test specimens. Other features include a small number of brittle second phase precipitates, including titanium rich and aluminium oxides, identified by EDX analysis, as well as a Laves phase, which was previously identified in the as-sintered microstructure.

The microstructure and mechanical properties of HIP Alloy 718 powder, which was then subsequently heat-treated, were investigated by Appa Roa et al. [4]. A homogeneous microstructure was produced with a fine grain size along with the presence of prior particle boundaries. The mechanical properties of the HIP and heat-treated sintered material were comparable with wrought materials. Fracture surfaces revealed a transgranular ductile mode of fracture in the solution annealed condition with an increased incidence of intergranular fracture by decohesion of prior particle boundaries in the aged condition, which also significantly reduced the ductility.

Condition	**Tensile Test Temperature (°C)**	**0.2% Proof Stress (Mpa)**	**Ultimate Tensile Strength (Mpa)**	**Elongation (%)**
MIM As-Sintered	20	503	936	20
MIM Sintered (Heat Treated)	20	1046	1211	6
MIM Sintered (Heat Treated)	540	895	1027	4
Cast (Heat Treated) †	20	915	1090	11
Wrought (Heat Treated) †	20	1185	1435	21
Wrought (Heat Treated) †	540	1065	1275	18

† ASM International Handbook Vol. 1.

Table 2: Mechanical properties of MIM master alloy Alloy 718.

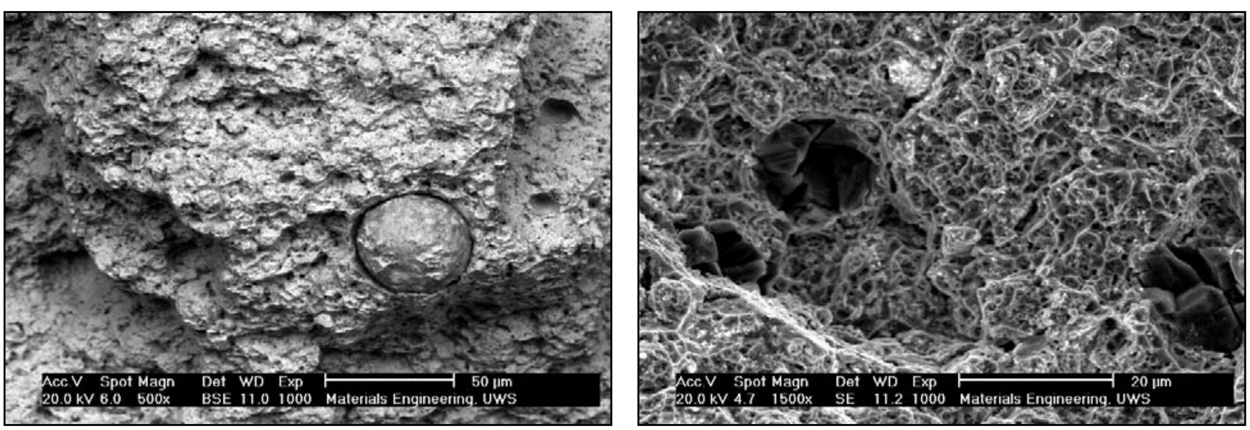

Figure 4: Fracture surfaces of MIM Alloy 718, featuring a prior particle and brittle second phase.

Valencia *et al.* [5] found that for MIM pre-alloyed Alloy 718, the best combination of mechanical properties and microstructure, with a reduced level of the deleterious Laves phase, achieved a 1238 MPA ultimate tensile strength. This was further improved to 1350 MPA with an additional HIP treatment. Tensile tests of HIP pre-alloyed Alloy 718 at elevated temperatures produce good results with an ultimate tensile strength of 1105 MPa at 540°C [6]. Previous results by Bose *et al.* [7] for MIM pre-alloyed Alloy 718 exceeded the minimum requirements for certain AMS mechanical property specifications. Earlier work indicated that initial fatigue and creep data also show promise, especially as a fine grain size is a major factor in achieving high fatigue strength [6, 7]. The distortion that occurred however during debinding and sintering may restrict future applications.

The sintering conditions were significantly different from the previous work where sintering was completed in vacuum with a partial pressure of argon, which is important to prevent chromium evaporation. Also the peak sintering temperature for the present work was lower and the time significantly shorter than MIM trials with pre-alloyed Alloy 718 powders. Further optimising of the sintering conditions and additional HIP with perhaps δ-phase heat treatments could further improve the present results. It is significant to note however that the master-alloy route provided good green strengths with little or no distortion observed after de-binding compared with previous results.

4. Conclusion

The work to date on MIM of Alloy 718 produced from the master alloy route shows some promise to provide economical net-shape components with superior properties to investment cast materials. The master alloy route produced a homogenous fine-grained microstructure with comparable mechanical properties to cast Alloy 718 and approaching the values of wrought Alloy 718, especially at high temperature (540°C). However, a full-scale investigation is required to fully validate the materials properties of MIM Alloy 718, in order to provide statistically valid mechanical properties, including fatigue and creep data, with the secondary aim of eradicating prior particle boundaries and deleterious second phases.

5. References

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