Influence of Gas Atomised Powder Size on Mechanical Properties of 15-5PH

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Abstract

Control of mechanical properties and surface roughness in MIM products is critical to their competitiveness against other fabrication methods. Material cost is also often cited as a barrier to further penetration of MIM technology. In this study we investigate the impact of starting particle size distribution on the sintering performance and finished properties of a precipitation-hardened stainless steel. The performance of conventional 90% -22µm powder is compared with coarser -32µm and -38µm powders which are prepared by sieving as opposed to classifying with consequent cost benefits. Rather than the more popular 17-4PH system, the 15-5PH alloy, which has a slightly lower Cu level, is investigated with a view to controlling Cu loss during furnacing and to moderating final hardness after furnace cooling. Results from 3-point bend and tensile testing of samples furnaced at different temperatures are presented alongside microstructural analysis. Commercial aspects of the use of coarser powders are discussed.

INTRODUCTION

Metal injection moulding (MIM) continues to grow in popularity as a means of producing large numbers of complex, precision parts for an increasing variety of industries [1,2]. Stainless steels remain the most important class of materials used in MIM and it is arguable that precipitation hardening steels are in turn the most widely used family of stainless alloys in MIM. They offer a combination of high strength, good corrosion resistance at relatively low cost (low Ni) and are relatively easy to sinter in a controlled manner. They usually require no post-sintering heat treatment but, owing to their high hardness, they can be difficult to finish to final dimensions if distortion occurs during furnacing.

Stucky et al [3] have examined the tendency to distortion in 17-4PH steels and the effect of using different powder feedstocks including gas atomised master alloys and prealloy powders and water atomised prealloy powders and combinations thereof. Their study concludes that a combination of gas atomised master alloys and prealloy powders can offer reduced distortion compared with other powder combinations.

17-4PH is the most widely used precipitation hardening alloy in MIM and among its potential drawbacks is the tendency for Cu evaporation during sintering. This can lead to the need for regular furnace cleaning to avoid cross contamination in some cases. It was partly with these aspects in mind, that the present study of 15-5PH was initiated. 15-5PH has a lower Cu level and also achieves lower hardness than the 17-4PH alloy. Nevertheless wrought properties typically quoted for 15-5PH are Ultimate Tensile Strength 1070MPa, %El 12%, R_c 39 in the H1025 heat treated state.

POWDER CHARACTERISTICS / 15-5 PH

The gas atomized 15-5PH powder used in this study was manufactured using proprietary gas atomisation technology, specifically designed to manufacture fine powder for MIM. A single batch of powder was produced by induction melting the raw materials in an inert atmosphere and atomising using nitrogen gas. The powder chemistry conforms to the 15-5PH specification as defined by the UNS designation S15500 as shown in Table 1.

Table 1. 15-5PH chemical specification and powder analysis.

Element	15-5PH Specification	Powder Analysis
Fe	Balance	Balance
Cr	14.0 - 15.5	15.30
Ni	3.5 - 5.5	4.49
Cu	2.5 - 4.5	3.64
Nb	0.15 - 0.45	0.38
Mn	1.00 max.	0.61
Si	1.00 max.	0.65
C	0.07 max.	0.04
P	0.04 max.	0.02
S	0.03 max.	<0.01

From the single batch of as-atomised powder, different fractions were extracted by air classification and sieving. Table 2 shows the particle size distribution characteristics d10, d50 and d90 of the powder feedstocks measured using a Malvern Mastersizer 2000. The three powder size distributions include an air classified product (90% -22µm) and two sieved powders (32, 38µm). Sieving was carried out using a high efficiency, ultrasonically-actuated sieve unit. Standard apparent and tap density measurements were also carried out on each product.

Table 2. Particle size distribution and powder density of starting materials.

Powder Size	D10 µm	D50 µm	D90 µm	Tap Density g/cc	Apparent Density g/cc
90% < 22 Microns	3.6	10.2	21.9	4.80	3.70
< 32 Microns	3.8	11.7	26.0	4.86	4.05
< 38 Microns	3.8	11.8	27.7	4.93	4.10

EXPERIMENTAL

Feedstock Fabrication and Component Fabrication

Three feedstocks of the different 15-5PH powders were compounded using a proprietary multicomponent binder system. A constant powder loading of 60 vol % obtained from rheological characterization was achieved. Compounding took place in a double-sigma compounder (Figure 1) under inert atmosphere.

Figure 1. Double-sigma compounder

Figure 2. Battenfeld HM 400 injection moulding machine

The feedstocks were then injection moulded using a Battenfeld HM 400 injection moulding machine (Figure 2). The mould for the tensile bars is shown in Figure 3.

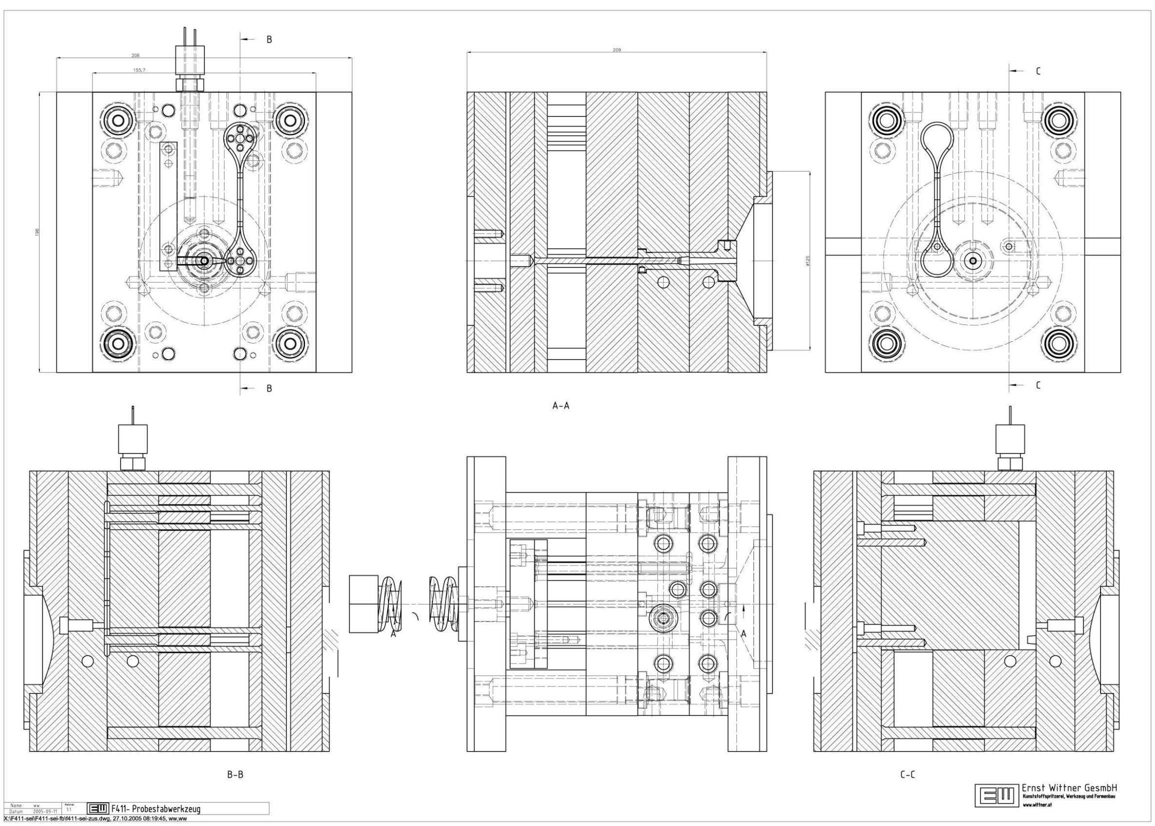


Figure 3. Mould for tensile bars and three-point bend bars

Figure 4. Green parts of 15-5 PH

Sintering of the green parts (Figure 4) took place in a Thermal Technology vacuum furnace at temperatures between 1320 ^{o}C and 1380^{o}C under hydrogen (Figure 5). The holding time at temperature was 3 hours followed by cooling at 10 ^{o}C / minute. Blaine et al [4] have determined that sintering of precipitation hardening stainless steels is enhanced in pure hydrogen compared with nitrogen/hydrogen atmospheres. They related this to the stabilisation of austenite by nitrogen which retards diffusion and densification compared with the δ-ferrite phase which otherwise forms.

Figure 5. Vacuum furnace and sintered parts

RESULTS

From each set of conditions which are listed in Tables 3 and 4, a number of the tensile bars were heat treated. The remaining samples were left in the non-heat treated condition after sintering.

Table 3. Parameter variation and nomenclature – samples before heat treatment

Sintering temp. / particle size	90.1% -22 μm	99.9% -32 μm	99.9% -38 μm
1320^{o}C	01A	02A	03A
1350^{o}C	01B	02B	03B
1380^{o}C	01C	02C	03C

Table 4. Parameter variation and nomenclature – samples after heat treatment

Sintering temp. / particle size	90.1% -22 μm	99.9% -32 μm	99.9% -38 μm
1320°C	01A-HT	02A-HT	03A-HT
1350°C	01B-HT	02B-HT	03B-HT
1380°C	01C-HT	02C-HT	03C-HT

From the non-heat treated tensile bars three bars for each condition were characterised using an AGC SLOW tensile test machine equipped with a Sandner 25 extensometer. A characteristic stress-strain diagram is shown in Figure 6.

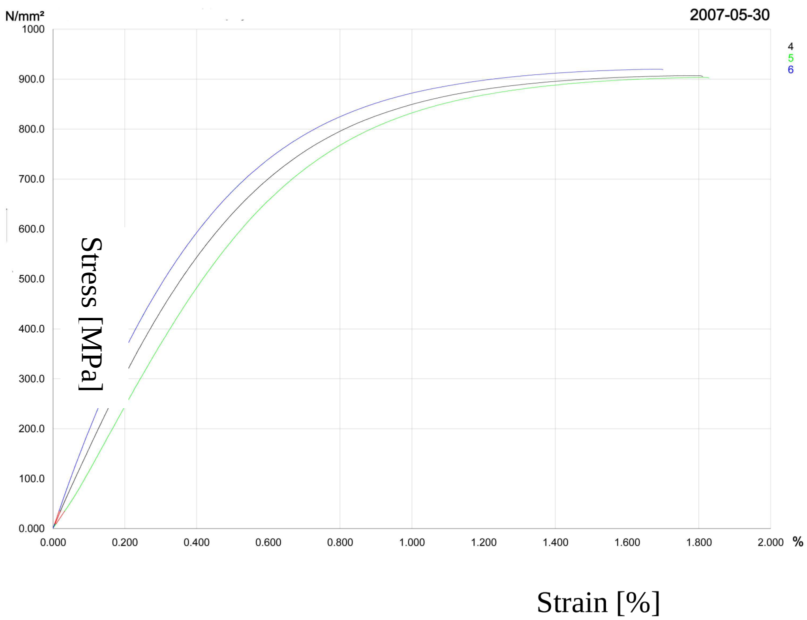


Figure 6 Stress-strain diagram of samples 01B

Table 5. Parameter variation: Ultimate Tensile Strength (UTS, R_m)[MPa] before heat treatment

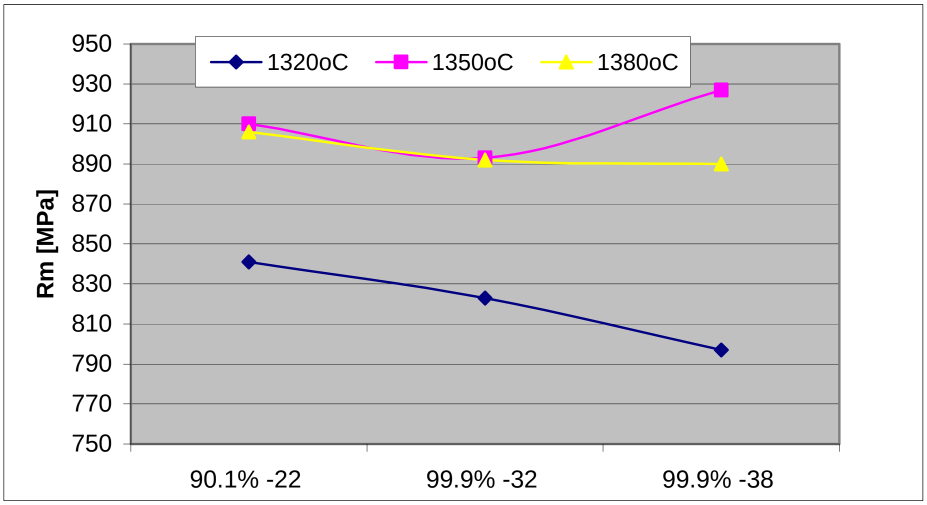
Sintering temp. / particle size	90.1% -22 μm	99.9% -32 μm	99.9% -38 μm
1320°C	841	823	797
1350°C	910	893	927
1380°C	906	892	890

Table 6. Parameter variation: Elongation A [%] before heat treatment

Sintering temp. / particle size	90.1% -22 μm	99.9% -32 μm	99.9% -38 μm
1320°C	1.0	0.92	0.62
1350°C	1.2	1.4	1.4
1380°C	2.4	2.0	2.3

The tensile properties in Tables 5 & 6 are presented in Figures 7 & 8 and demonstrate that UTS is highest at highest sintering temperatures (1350 °C, 1380°C) and that at 1320°C, UTS falls off as particle size becomes coarser. At the higher temperatures, there is little difference in UTS among the powder types.

The trends in ductility shown in Figure 8 are more discriminating in showing that again, low sintering temperature translates into low ductility. Furthermore, 1380^{o}C is shown to give superior ductility to 1350^{o}C. However, apart from the data set for 1320^{o}C, there is no marked trend in going from fine to coarse powders.



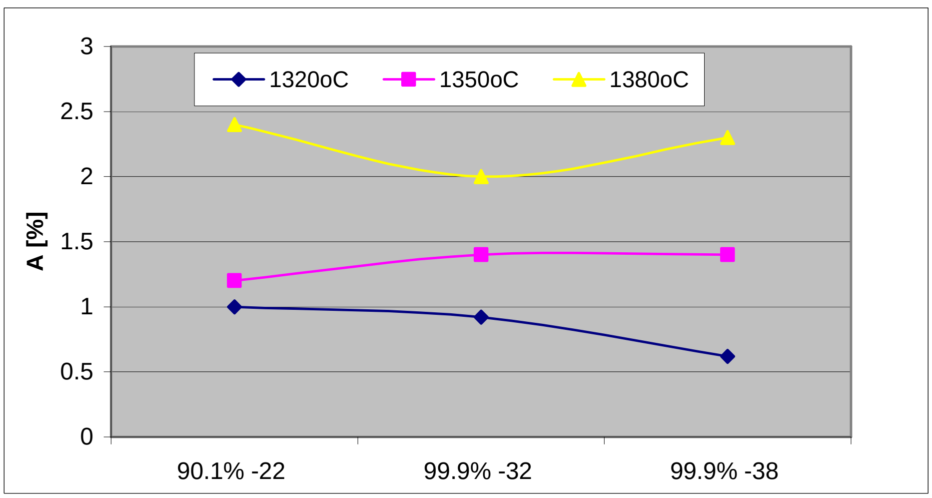


Figure 7. Effect of Sintering temperature on UTS (R_m) and Elongation (A%) of 15-5PH in the as-sintered condition.

DISCUSSION

The size distributions of each powder type (90% -22µm, -32µm and -38µm) presented in Table 2, show quite similar median sizes despite very different top cut off values. D50 only varies by 1.5µm but the D90 of the classified and sieved fractions vary across a 5.8µm range. There is a clear relationship between both apparent and tap density and the powder size distribution. Finer powders with narrower size distributions tend to show lower density due to increased inter-particle friction and inferior particle packing.

This difference in particle packing is also in evidence in the feedstocks produced for injection moulding. Nevertheless, all samples moulded well and showed good green properties. Lee et al [5], determined that binder and feedstock formulation usually need to be adjusted according to size of powders in order to achieve adequate melt flow index and injection performance but this was not the case in this study. Today, MIM powders are available in sizes from 80% -5µm (for MicroMIM) through e.g. 90% -10µm, 90% -16µm, 90% -22µm, 80% -22µm and sizes for different applications are selected based on required precision, surface finish, density, mechanical properties and cost. Physical size of the component to be moulded and hence mould design and gate geometry will ultimately have some say on the maximum particle size that can be tolerated.

The results here show that for all powder sizes, a sintering temperature of at least 1350°C is required to achieve best UTS and that even higher temperature gives that combined with best ductility. This is believed to point to improved densification at higher temperature although the concern if too high a temperature is used is that Cu loss will be exacerbated. Experience elsewhere is that in order to achieve high density, sintering temperatures in the range 1250-1390°C are required. In absolute terms, the strength levels and ductility fall short of wrought properties reported in the introduction. Further work, not reported here [6], examines the effect of heat treatment on properties.

Regarding the impact of particle size distribution, then it appears that above 1350°C there is little to choose among the finest to the coarsest products in terms of mechanical properties. While the amount of testing to date has been limited, and indeed results from heat treated samples are not yet available, there is a good indication that acceptable properties can be achieved with somewhat coarser powder grades than have been conventionally favoured for MIM. Of course the surface finish, which has not been measured, may show that the finer product offers advantages that determine it will remain the favoured option, but for applications where mechanical properties and cost are key drivers, then coarser powders may provide a partial solution.

The cost benefit associated with coarser sieved powders is two-fold. First, the transition from an air classified product to a sieved product increases the powder yield significantly. Second, the operating costs of a classifier are higher than a sieving operation. Furthermore, the capital cost of a sieve is far less than that of an air classification unit.

SUMMARY & CONCLUSIONS

Gas atomised 15-5PH powders of three different sizes have been produced by air classification and sieving. Preliminary mechanical testing shows that provided a sintering temperature above 1350°C is used, there appears little difference in properties achieved for the different particle sizes. Therefore in applications where cost is critical and mechanical properties dictate material selection, there is scope to evaluate sieved powders for MIM which might previously have been thought too coarse to consider.

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