

# 6

## AN INFLUENCE OF THE SS316L POWDER PARTICLE SHAPE TO THE DENSIFICATION OF METAL INJECTION MOLDING (MIM)

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### 6.0 INTRODUCTION

Metal injection molding (MIM) has acquired increasing importance as a production technique for small, complex stainless steel components [1, 2]. Sintering is critical for determining the final quality of the parts produced by MIM. Because high sintered density is imperative for good mechanical properties and corrosion resistance, achieving full or near-full density has been a major objective of sintering [3]. Therefore, most research on 316L stainless steel sintering to date has focused on the sintering behavior of the molded parts especially for gas-atomised powder in argon environment [3-6]. An understanding of the factors

influencing densification of stainless steels is important as over 50% of the injection molded and sintered components are made from stainless steel compositions [7].

In a metal injection molding (MIM) process, gas-atomised powder is generally used due to their high packing density and associated feedstock rheology. The sintered components exhibit mechanical and corrosion properties similar or superior to that of wrought material. Water-atomised powders in MIM can be economical and have an improvement in shape retention during debinding and sintering. However, their use comes with a penalty of lower powder loading and sintered density, with a corresponding degradation in the mechanical and corrosion properties. Studies reveal that injection molded and sintered components using water-atomised 316L stainless steel powders have a residual porosity of 3–5% for similar particle characteristics and sintering conditions as that of gas-atomised powders [5]. This article investigates a densification of SS316L gas and water-atomised compact sintered in high vacuum environment at temperature ranging from 1340 to 1400 °C.

## **6.1 METHODOLOGY**

MPIF 50 standard tensile bar is used as a specimen. A 316L stainless steel gas and water-atomised powder with pycnometer density of 7.99 g/cm<sup>3</sup> and 7.90 g/cm<sup>3</sup> respectively are mixed with 73 % PEG weight of polyethylene glycol (PEG) and 25 % weight of polymethyl methacrylate (PMMA). About 2 % weight of stearic acid (SA) is used as a surfactant. Powder particles characteristics used in the experiment are shown in Table 1 and the measurement was measured by Mastersizer, Malvern Instrument.

Prior to the injection, compositions are mixed in a sigma blade mixer for 95 minutes at of 70°C. Battenfeld, BA 250 CDC injection molding machine was used to prepare the greens while

high vacuum furnace Korea VAC-TEC, VTC 500HTSF with vacuum pressure up to  $9.5 \times 10^{-6}$  mbar was used for sintering.

**Table 1** Powder particles characteristic

a) Gas-atomised powder

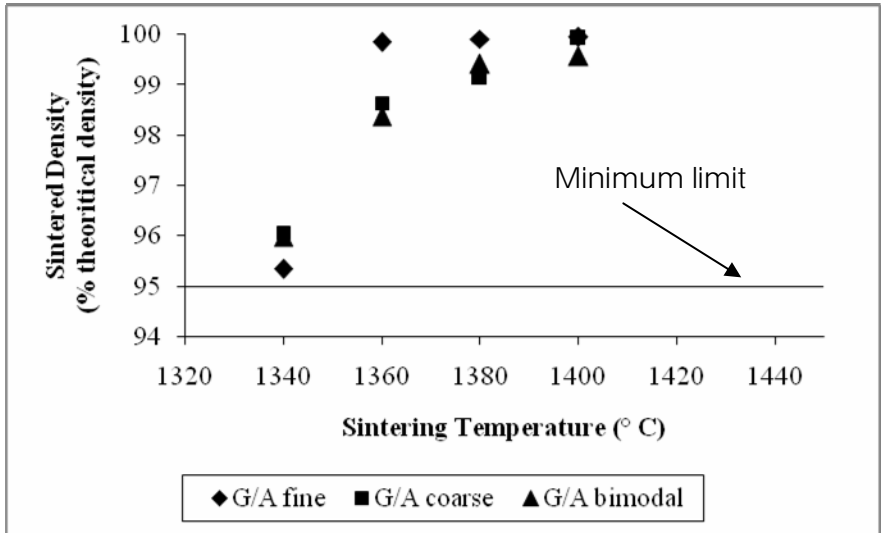
	repeat	D <sub>10</sub>	D <sub>50</sub>	D <sub>90</sub>	S <sub>w</sub>	S (m <sup>2</sup> /g)
Coarse	1	9.228	19.456	45.696	3.685	0.145
	2	9.972	19.586	36.435	4.549	0.144
	mean	9.600	19.521	41.066	4.117	0.144
Fine	1	6.080	11.130	17.800	5.488	0.617
	2	5.480	11.320	21.880	4.258	0.622
	mean	5.780	11.225	19.840	4.873	0.619

b) Water-atomised powder

	repeat	D <sub>10</sub>	D <sub>50</sub>	D <sub>90</sub>	S <sub>w</sub>	S (m <sup>2</sup> /g)
Coarse	1	5.216	15.748	34.989	3.097	0.551
	2	4.746	14.228	34.667	2.964	0.599
	3	4.994	15.179	34.585	3.046	0.570
	mean	4.985	15.052	34.747	3.036	0.573
Fine	1	3.301	6.946	15.251	3.852	1.000
	2	3.331	7.121	16.866	3.634	0.982
	3	3.382	7.403	20.429	3.278	0.946
	mean	3.338	7.157	17.515	3.588	0.978

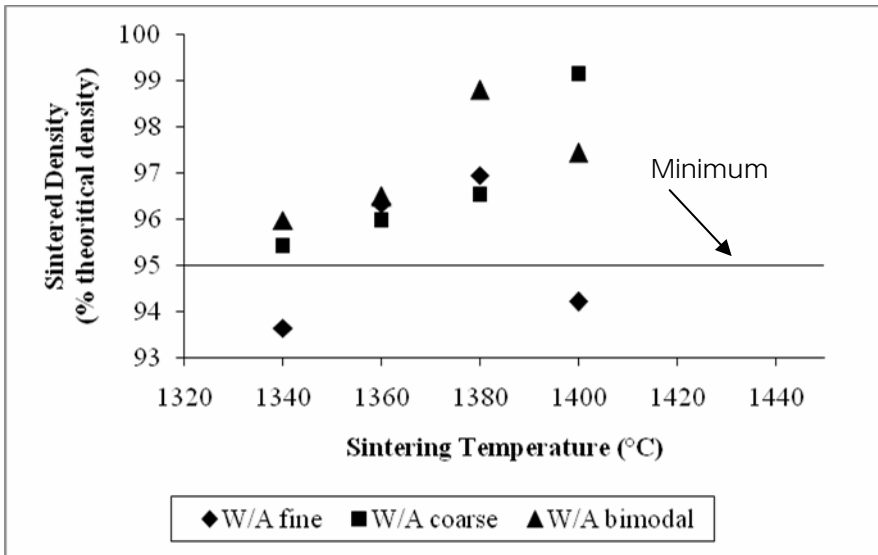
## RESULTS & DISCUSSION

Figure 1 and 2 demonstrate that the sintered density has increased gradually when the sintering temperature increases. In this chapter, the heating rate remains at 10 °C/minute. This is based on earlier study published by Mohd Afian Omar [4] and Suri et al. [5] that such heating rate is sufficient for sintering SS316L compact. Prior to reaching the sintering temperature, pre-sintering was performed at 600 °C in 20 minutes under the same atmosphere to decompose remaining binders left by thermal pyrolysis [8].



**Figure 1**

Gas-atomised compact sintered density. Heating rate and cooling rate: 10 °C/minit; thermal pyrolysis: 600 °C for 20 minutes; dwell time: 4 hour



**Figure 2** Water-atomised compact sintered density. Heating rate and cooling rate: 10 °C/minit; thermal pyrolysis: 600 °C for 20 minutes; dwell time: 4 hour

The minimum limit shown in Figure 1 and 2 refers to the minimum MIM compact density suggested by German and Bose [7]. In Figure 1, sintered density of gas-atomised compact exceeds the minimum limit at sintering temperature from 1340 to 1360 °C. However, fine powder compact demonstrates better densification than coarse powder compact, except bimodal powder compact is less dense than the monomodal powder. The phenomenon shown in Figure 1 is almost the same as obtained by Mohd Afian Omar [4] where fine powder compact obtained better sintered density than the coarse powder compacts as well as bimodal powder compact. This is due to larger surface area,  $S$  for fine powder particles as shown in Table 1 where fine gas-atomised powder has larger surface area than the coarse powder which will promote faster densification [9]. In addition, sintered density obtained in

this chapter is higher than obtained by Mohd Afian Omar's data [4], which utilizes argon atmosphere for sintering the same powder compact. At 1360 °C, the sintered density obtained by Mohd Afian Omar [4] was only 97 % of the theoretical density (fine powder compact) while the sintered density obtained in this chapter is 99.86 % of the theoretical density after sintering in the high vacuum atmosphere. Moreover, coarse powder compact also shows higher sintered density (98.64 % of the theoretical density) compared to Mohd Afian Omar [4] where only 95.3 % of the theoretical density was obtained.

In addition, this chapter attains better density for bimodal gas-atomised particle size distribution compact. The obtained sintered density is 98.37 % of the theoretical density compared to German [12] where only 83 % of the theoretical density sintered was obtained for 1 hour in argon environment, while Mohd Afian Omar [4] obtained 97.5 % of the theoretical density when sintered for 4 hour also under the same environment as German [12]. An improvement of sintered density presented in this chapter is mainly contributed by the vacuum environment and it has been confirmed by Ji et al. [11]. Mohd Afian Omar [4] found that fine powder in a coarse powder matrix will improve sintered density. This is due to the fact that fine powder has improved particles surface area and thus increases energy reduction rate between particles, thus enhances densification.

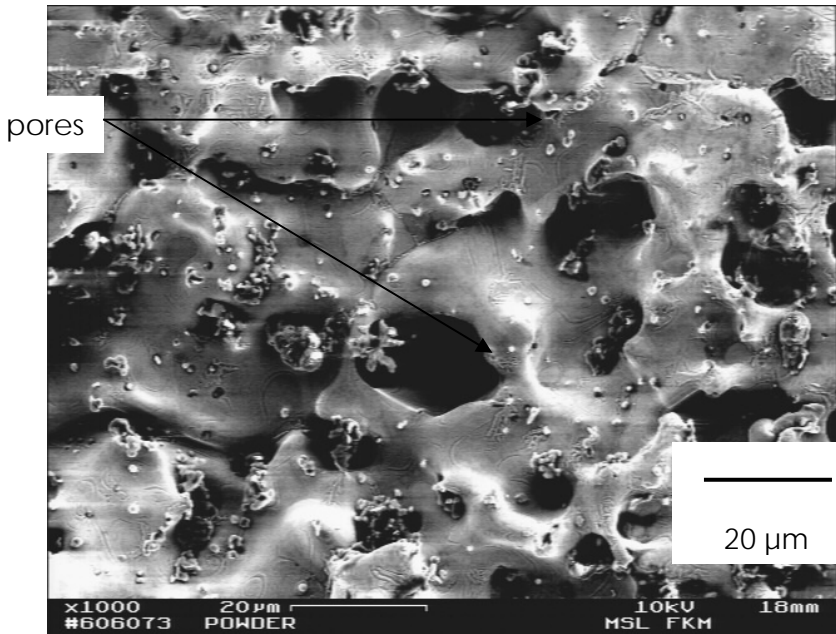
Furthermore, Figure 2 demonstrates that fine water-atomised compact is unable to achieve minimum sintered density when sintered at 1340 °C, for the reason of the irregular shape of the water-atomised powder particles compared to the gas-atomised powder. Besides that, more inter-particle friction occurred on the water-atomised powder as the surface area,  $S$  of the fine water-atomised powder is larger than the gas-atomised powder (Table 1), contributing to a low critical powder loading and thus the optimum powder loading for this powder is limited to 62.5 % volume. On the other hand, bimodal water-atomised compact stand out in the sintered density aspect compared to the monomodal compacts. Bimodal compacts attain highest sintered density at sintering

temperature 1340 °C, 1360 °C and 1380 °C. However, a vice versa situation occurred at 1400 °C. Sintered density of a fine powder compact has plummet from 96.96 % to 94.21 % of theoretical density while sintered density of water-atomised bimodal compact also slumped from 98.81 % to 97.44 % of the theoretical density when sintering temperature rose to 1400 °C. Nevertheless, sintered density of the coarse powder compact remains high at 99.14 % of the theoretical density.

Reduction of sintered density occurred due to excess of liquid phase in the powder matrix during sintering at 1400 °C, especially when the powder loading is too low in fine water-atomised powder compact. Liquid phase is expected to exist in SS316L when the sintering temperature reaches 1350 °C and 1390 °C [8]. Melting temperature of SS316L is 1375 °C [10]. However, liquid phase in solid will enhance the densification process, but too much liquid will reduce sintered density due to micro structure coarsening [11]. Particle melting occurs during liquid phase sintering, resulting in a solid-liquid mixture during the thermal cycle. The liquid phase provides bonding, contributes a capillary force, and usually enhances the rate of mass transport as compared to solid-state process. Furthermore, coarse powder compact sintered at 1400 °C is able to improve its density to 99.14 % of theoretical density. This is due to the small surface area,  $S$  that eliminates liquid phase in the compact. Small surface area of powder particles delays the liquid phase formation although the sintering temperature is already exceeding materials melting temperature. This occurs by the fact that less interparticle contact due to small surface area delay the surface energy reduction.

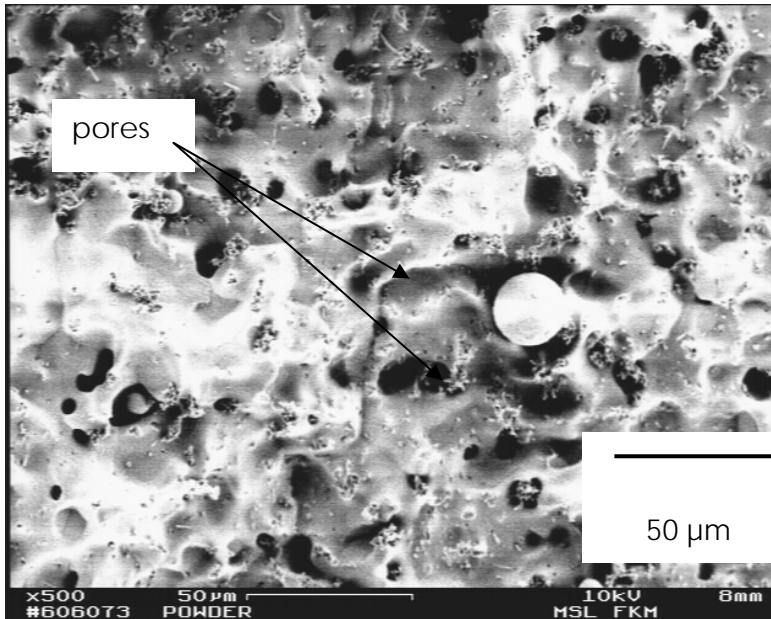
Figure 3 and 4 shows the SEM image of fine water-atomised and fine gas-atomised powder compact respectively sintered at 1360 °C. Both figures demonstrate that the liquid phase has appeared in the solid matrix during sintering. This is shown by the SEM image where most powders have melt especially for the gas-atomised compact (Figure 4), resulting to better density for the gas-atomised over water-atomised powder compact. This is caused by the regular shape of the gas-atomised powder which has larger

interparticle contact which in turn enhances surface energy reduction by reducing surface area by concomitant forming of interparticle bonds. Less porosity appeared in the gas atomised compact as shown in Figure 4, which results to better sintered density over the water atomised compact shown in Figure 3.



**Figure 3** Scanning electron micrograph of water-atomised compact sintered at 1360 °C. Sintered density: 96.34 % of theoretical density





**Figure 4** Scanning electron micrograph of gas-atomised compact sintered at 1360 °C. Sintered density: 99.86 % of theoretical density

## CONCLUSION

- Sintering in vacuum atmosphere offers better densification compared to sintering in gas atmosphere as proven by Ji et al. 2001 [11].
- Fine gas-atomised powder compact demonstrates better densification than the coarse powder compact except for bimodal powder compact which is less dense than the monomodal gas-atomised powder.
- Bimodal gas-atomised particle size distribution compact attains better densification compared to the results reported by German [10] and Mohd Afian Omar [4].

- Bimodal water-atomised compact stands out in the sintered density property compared to the monomodal compacts except at sintering temperature, 1400 °C.
- Sintered density of fine and bimodal water-atomised powder compact plummet when sintered at 1400 °C.

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