DENSIFICATION OF SS316L GAS-ATOMIZED AND WATER-ATOMIZED POWDER COMPACT

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ABSTRACT

Sintering is a key step in the metal injection molding process, which affects the final density as well as the mechanical properties of the sintered part. Densification of metal injection molded 316L stainless steel is one of the hottest research area in recent years. The present study investigates a densification of 316L stainless steel in order to understand the differences in the densification behavior of metal injection molded gas-and-water-atomized powder. Densification of monomodal particle size distribution consisting of fine and coarse powder, as well as bimodal particle size distribution powder for both gas-and-water-atomized SS316L is investigated with sintering temperature ranging from 1340 to 1400 °C while heating rate and dwell time remains at 10 °C/min and 4 hour respectively. At the mean time cooling rate was kept the same as the heating rate. Results show that both gas-and-water-atomized powders could be sintered to a maximum of 99 % of theoretical density, which is very close to SS316L's solid density.

Keywords: Sintering, gas-atomized powder, water-atomized powder, 316L stainless steel

INTRODUCTION

Metal injection molding (MIM) has acquired increasing important as production technique for small, complex stainless steel components (Heaney 2004; Heaney et al. 2004). 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 (Hezhou et al. 2008). Therefore, most research on 316L stainless steel sintering to date has focused on the sintering behavior of the molded parts especially for gas-atomized powder in argon environment (Mohd Afian Omar 1999; Suri et al. 2005; Koseski et al. 2005; Hezhou et al. 2008). 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 (German & Bose 1997).

In a metal injection molding (MIM) process, gas-atomized 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-atomized powders in MIM can be economical and improve 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-atomized 316L stainless steel powders have a residual porosity of 3–5% for similar particle characteristics and sintering conditions as that of gas-atomized powders (Suri et al. 2005). This article investigates a densification of SS316L gas and water-atomized compact sintered in high vacuum environment at temperature ranging from 1340 to 1400 °C.

METHODOLOGY

MPIF 50 standard tensile bar is used as a specimen. A 316L stainless steel gas and water-atomized powder with pycnometer density of 7.99 g/cm^3 and 7.90 g/cm^3 respectively is 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 characteristic used in the experiment is 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 a temperature of 70^{o}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 9.5×10^{-6} mbar was used for sintering.

Table 1 Powder particles characteristic

a) Gas-atomized powder

	repeat	D_{10}	D_{50}	D_{90}	S_w	S (m^2/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-atomized powder

	repeat	D_{10}	D_{50}	D_{90}	S_w	S (m^2/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 demonstrates that the sintered density has increased gradually when the sintering temperature increases. In this study, the heating rate remains at 10 °C/minute. This is based on earlier study published by Mohd Afian Omar (1999) and Suri et al. (2005) that such heating rate is sufficient for sintering SS316L compact. Prior sintering temperature is reached, pre-sintering was performed at 600 °C in 20 minutes under the same atmosphere to decompose remaining binders left by thermal pyrolysis (Li et al. 2003).

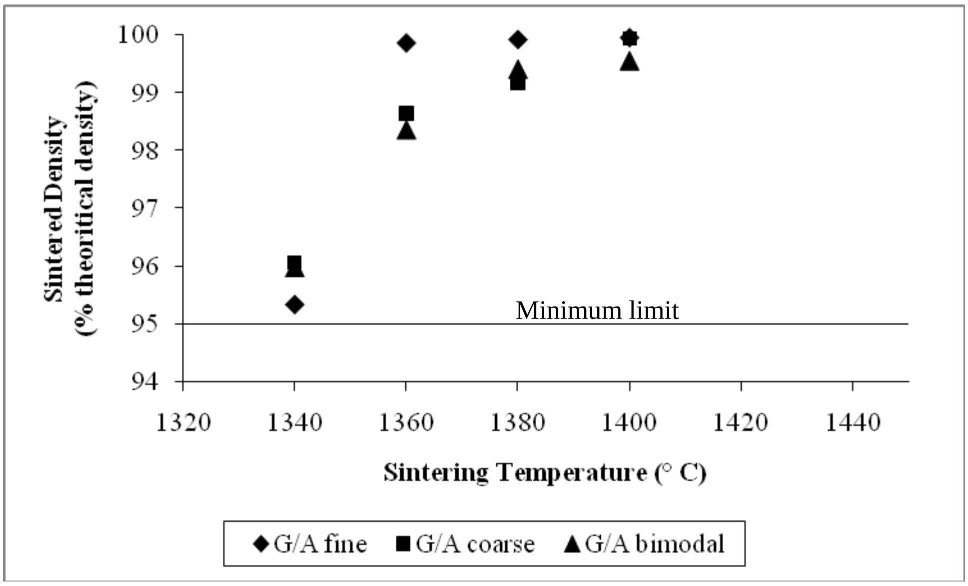

Figure 1 Gas-atomized compact sintered density. Heating rate and cooling rate: 10 °C/minit; thermal pyrolysis: 600 °C for 20 minutes; dwell time: 4 hour

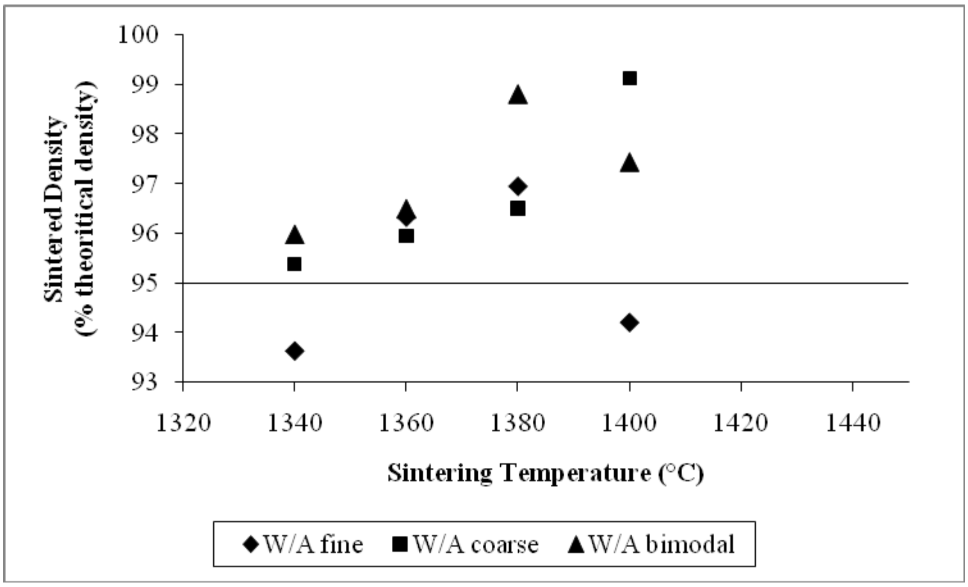

Figure 2 Water-atomized compact sintered density. Heating rate and cooling rate: 10 °C/minit; thermal pyrolysis: 600 °C for 20 minutes; dwell time: 4 hour

The minimum limit shown in Figure 1 and 2 refer to the minimum MIM compact density suggested by German and Bose (1997). In Figure 1, sintered density of gas-atomized 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. Phenomenon shown in Figure 1 is more less the same as obtained by Mohd Afian Omar (1999) that 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 owing by fine powder particles as shown in Table 1 that fine gas-atomized powder has larger surface area than the coarse powder that will promote faster densification (Kang 2005). In addition, sintered density obtained in this study is higher than obtained by Mohd Afian Omar (1999), which utilizes argon atmosphere for sintering the same powder compact. At 1360 °C, the sintered density obtained by Mohd Afian Omar (1999) was only 97 % of the theoretical density (fine powder compact) while the sintered density obtained in this study 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 (1999) only 95.3 % of the theoretical density.

In addition, this study attains better density for bimodal gas-atomized particle size distribution compact. Sintered density obtained is 98.37 % of the theoretical density compared to German (1992) only 83 % of the theoretical density sintered for 1 hour in argon environment, whilst Mohd Afian Omar (1999) obtained 97.5 % of the theoretical density when sintered at for 4 hour also under the same environment as German (1992). An improvement of sintered density presented in this study is mainly contributed by the vacuum environment and it has been confirmed by (Ji et al. 2001). Mohd Afian Omar (1999) found that fine powder in a coarse powder matrix will improve its sintered density. This is by 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 fine water-atomized compact is unable to achieve minimum sintered density when sintered at 1340 °C, for the reason that the irregular shape of the water-atomized powder particles compared to the gas-atomized powder. Beside that, more inter-particle friction occurred on the water-atomized powder as a surface area, S of the fine water-atomized powder is larger than the gas-atomized 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. At the mean time bimodal water-atomized compact dominates the sintered density 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-atomized bimodal compact also slump 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 the fine water-atomized powder compact. Liquid phase is expected to exist in SS316L when the sintering temperature reaches 1350 °C and 1390 °C (Li et al. 2003). Melting temperature of SS316L is 1375 °C (German 1996).

However, liquid phase in solid will enhance the densification process, but too much liquid will reduce sintered density due to micro structure coarsening (Ji et al. 2001). 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 the fine water-atomized and fine gas-atomized powder compact respectively sintered at 1360 °C. Both figures demonstrate that the liquid phase has been appeared in the solid matrix during sintering. This is shown by the SEM image that most powders is melt especially on the gas-atomized compact (Figure 4), resulting better density of the gas-atomized over the water-atomized powder compact. This is caused by the regular shape of the gas-atomized powder having larger interparticle contact, enhances surface energy reduction by reducing surface area with concomitant formation of interparticle bonds. Less porosity appeared in the gas atomized compact as shown in Figure 4, results better sintered density over the water atomized compact shown in Figure 3.

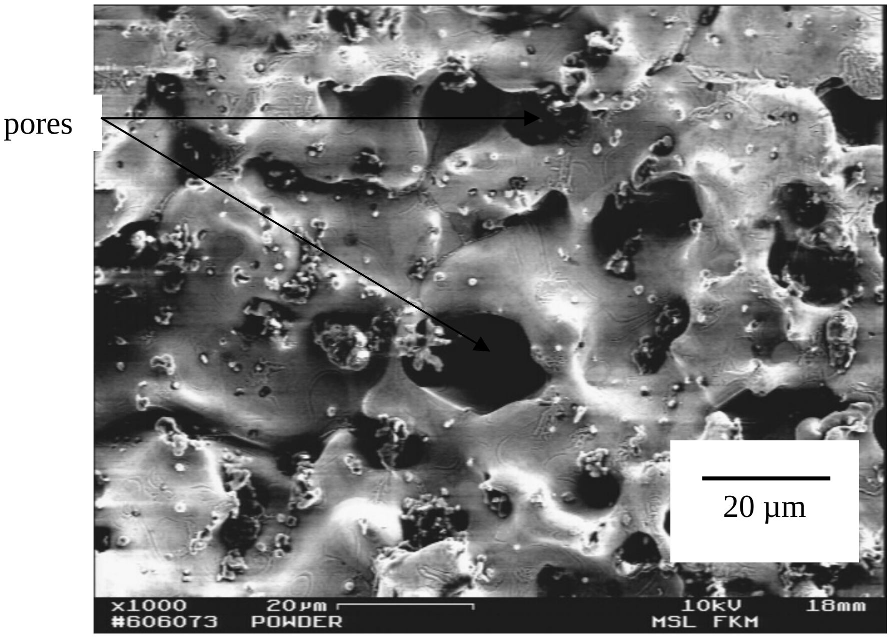

Figure 3 Scanning electron micrograph of water-atomized compact sintered at 1360 °C. Sintered density: 96.34 % theoretical density

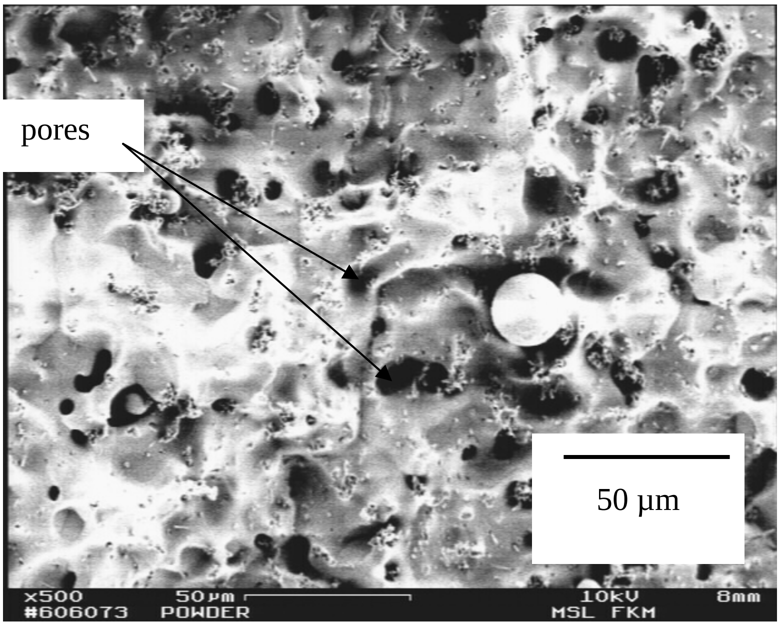

Figure 4 Scanning electron micrograph of gas-atomized compact sintered at 1360 °C. Sintered density: 99.86 % theoretical density

CONCLUSIONS

- Sintering in vacuum atmosphere offer better densification compared to sintering in gas atmosphere as it has been proved by (Ji et al. 2001).
- Fine gas-atomized powder compact demonstrates better densification than the coarse powder compact except bimodal powder compact is less dense than the monomodal gas-atomized powder.
- Bimodal gas-atomized particle size distribution compact attain better densification than reported by German (1992) and Mohd Afian Omar (1999).
- Bimodal water-atomized compact dominates the sintered density compared to the monomodal compacts except at sintering temperature 1400 °C.
- Sintered density of a fine and bimodal water-atomized powder compact is plummeting when sintered at 1400 °C.

REFERENCES

German, R.M. 1992. Prediction of sintered density for bimodal powder mixtures. *Metallurgical Transactions A.* 23A: 1455-1465.

German, R.M. 1996. Sintering Theory and practice. A Wiley-Interscience Publication. USA

Heaney, D.F, Mueller, T. & Davies, P. 2004. Mechanical Properties of Metal Injection Molded 316L Stainless Steel Using Both Prealloy and Master Alloy techniques. *Journal of Powder Metallurgy*. 47(4): 367-373.

Heaney, D.F. 2004. Qualification Method for Powder Injection Molded Components. *P/M Science & Technology Briefs*. 6(3): 21-27.

Hezhou, Y., Liu, X. Y. & Hong H. 2008. Sintering of 17-4PH stainless steel feedstock for metal injection molding. *Materials Letters*: 62:3334–3336.

Ji, C.H., Loh, N.H., Khor, K.A. & Tor, S.B. 2001. Sintering study of 316L stainless steel metal injection molding parts using Taguchi method: final density. *Materials Science and Engineering A*. 311:74–82.

Kang, S.J.L. 2005. Sintering Densification, grain growth and microstructure. Elsevier Butterworth-Heinemann. UK

Koseski, R. P., Suri, P., Earhardt, N. B., German, R.M. & Kwon, Y. S. 2005. Microstructural evolution of injection molded gas-and water-atomized 316L stainless steel powder during sintering. *Materials Science and Engineering A*. 390: 171–177.

Li, S., Huang, B., Li, D., Li, Y., Liang, S. & Zhou, H. 2003. Influence of sintering atmosphere on densification process of injection molded gas atomized 316L stainless steel. *Powder Metallurgy*. 46 (3): 241-245.

Mohd Afian Omar. 1999. Injection molding of 316L stainless steel and NiCrSiB alloy powders using a PEG/PMMA binder. Ph.D. Thesis University of Sheffield, UK.

German R.M. & Bose, A. 1997. Injection Molding of Metals and Ceramics. Metal Powder Industries Federation, Princeton. NJ

Suri, P., Koseski, R.P. & German, R.M. 2005. Microstructural evolution of injection molded gas- and water-atomized 316L stainless steel powder during sintering. *Materials Science and Engineering A*. 402:341–348.