# SINTERING PARAMETER OPTIMISATION OF THE SS316L METAL INJECTION MOLDING (MIM) COMPACTS FOR FINAL DENSITY USING TAGUCHI METHOD

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# ABSTRACT

Sintering parameters of the SS316L water atomised injection moulded compact has been optimized for its best sintered density. The  $L_9$  (3<sup>4</sup>) Taquchi orthogonal array is used in the experiment while sintering temperature, sintering time, heating rate and cooling rate was selected as factors that influenced the sintered density. The sintering environment was in the vacuum and four replications were done for each trial. The analysis of variance shows that the confident level for the experiment was 99.5 % ( $\alpha = 0.005$ ) and all factors are highly significant at  $\alpha = 0.005$  to the sintered density. The study concluded that the heating rate has the highest influence to the sintered density (41.29 %) followed by sintering temperature (31.60 %), sintering time (11.13 %) and cooling rate (11.10%). The optimum sintered density obtained is 98.48 % of the theoretical density and the optimum parameter has been verified that the sintered density obtained is in a range of confident interval.

Key words: Sintering; Stainless steel; Taguchi method; Metal injection moulding; Powder injection moulding

# 1. INTRODUCTION

Metal injection moulding (MIM) is a relatively new processing technology used in powder metallurgy processing industries. This process is especially costeffective and beneficial for manufacturing small and complex components in large quantities. Metal injection moulding is used in an increasing range of different fields, including automotive, medical and telecommunications industries. It includes four basic steps consisting of mixing the powders and binders, injection moulding, debinding and finally sintering. Both injection moulding and sintering are the most important steps related to forming the green part and the final part respectively.

Therefore, an optimisation of the processing parameter is essential to obtain high quality final part. High sintered density of the final part is vital to maintain an excellent performance of the powder metallurgy products. Many earlier studies about sintering of MIM part are concerning with microstructures, densification, and sintering atmosphere (Li et al. 2003; Fu et al. 2005; Koseski et al. 2005; Suri et al. 2005; Bergine et al. 2006). Sintering parameters were optimised by adjusting the sintering variables without using any design of experiment (DOE) methodology. The traditional experimental approach that vary one variable at a time, holding all other variables as fixed does not produce satisfactory results in a wide range of experimental settings. Thus it requires a lot of experiments attempt before the optimised sintering parameter is obtained without having any statistical confidence level.

DOE for the injection parameter has been studied by Khairur Rijal Jamaludin et al. (2008a, 2008b) and Jamaludin et al. (2008) resulting a significant optimum injection parameter for MIM feedstock. As a consequence to the injection parameter optimised by those literatures, this paper presents a sintering parameter optimisation which utilises the optimised injection parameter. In addition, study by Ji et al. (2001) has shown the significance of sintering variables such as heating rate, dwell time, sintering temperature and sintering atmosphere. Ji et al. (2001) found that the vacuum environment is best for sintering SS316L and thus, this study attempts to continue the study by using a high vacuum environment in the experiment. Despite cooling rate is replacing the sintering environment in the orthogonal array, heating rate, dwell time and sintering temperature remains as sintering variables for the optimisation. These variables can influence the microstructure, pore size and shape and final density of the sintered parts.

## 2. EXPERIMENTAL

MPIF 50 standard tensile bar is used as a specimen. A water atomised 316L stainless steel powder with  $D_{50}$  of 7.157  $\mu$ m, pycnometer density of 7.90 g/cm<sup>3</sup> 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.

Prior to the moulding, compositions are mixed in a sigma blade mixer for 95 minutes at a temperature of 70°C. Battenfeld, BA 250 CDC injection moulding 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.

# 3. DESIGN OF EXPERIMENT

There are many sintering parameters that have some effect on the properties of the sintered density. Therefore, a design of experiment (DOE) methods is necessary for the experimental work involving many inputs. The most frequently used methods are partial or full factorial design and the Taguchi approach. With an appropriate DOE, one can quickly and with fewer amounts of trials, found whether the variables have an effect on the output quality. The Taguchi approach is mostly used in the industrial environment, but it can also be used for scientific research. The method is based on balanced orthogonal arrays (Park 1996). In this paper,  $L_9$  (3<sup>4</sup>) orthogonal array consisting of 9 experiment trials and 4 column is used as DOE followed by ANOVA to determine the significant level and contribution of each variables to the sintered density. The main variables involved in this study are as shown in Table 1. Three levels for each variable refer to the maximum and minimum limit that influences sintered density.

Table 1: Factor level (variables) in the experiment

	Factor		Level	
	racio	0	1	2
A	Sintering Temperatu re (°C)	1340	1360	1380
в	dwell time (minute)	60	120	240
С	Heating rate (°C/min)	6	8	10
D	Cooling rate (°C/min)	6	8	10

#### 4. RESULTS & DISCUSSION

The density of the sintered part was measured by Archimedes immersion method according to the MPIF 42. Four replications were recorded for each experiment as shown in Table 2. The theoretical density shown in Table 2 is calculated from the average of the replication. As shown in Table 2, a combination of A1 B2 C0 D1 results a maximum sintered density (98.48 % of the theoretical density) while, a combination of A2 B0 C2 D1 produce a minimum sintered density (93.53 % of theoretical density). Although the sintering temperature is only 1360 °C, slow heating rate (6 °C/minutes) and longer dwell time (240 minutes) enables the compact to obtain a maximum sintered density. Nevertheless, with high sintering temperature (1380 °C) and quick heating rate at shorter dwell time will minimise the sintered density.

Beside that, as shown in Table 3 is the analysis of variance (ANOVA) which demonstrates the significance level of the variables as well as the effect of the sintering variables to the sintered density. Generally, all the sintering variables have significant effect on the sintered density at 99.5 % significant level. The significant level obtained by this experiment is higher than reported by Ji et al. (2001). The ANOVA in Table 3 display the relative significance of the variables as well as the contributions of the variables assigned to the orthogonal array shown in Table 2. The ANOVA in Table 3 depict a very significant level ( $\alpha =$ 0.005) of each variables. Heating rate (C) is the most influential (41.29 %) to the sintered density, followed by the sintering temperature (A), dwell time and cooling rate. However, Ji et al. (2001) reported that the sintering atmosphere has been the most significant variable to the sintered density as it demonstrates the much higher variance ratio, F. The sintering atmosphere is the most influential variable (76.685 %)

followed by heating rate (7.377 %), sintering temperature (5.538%) and dwell time (5.168 %). Thus, based on his study, a high vacuum sintering environment has been considered. Beside that, the influence of cooling rate to the sintered density is investigated as this variable has not been studied by Ji et al. (2001) in his DOE. This is by the fact that the cooling rate is another sintering variable (Kang 2005). Although cooling rate is one of the sintering variables, it has been demonstrating a lowest contribution percentage. Despite the contribution is low, the high significant level,  $\alpha$  as shown by Table 3 is still indicate the importance of this variable. This is as important as dwell time which has been reported less influential by Ji et al. (2001) to the sintered density.

Table 2: Orthogonal array an	d sintered	density
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		Replication (density(g/cm <sup>3</sup> ))									
	Trial	A	В	С	D	1	2	3	4	Ÿ	% Theoretical density
	1	0	0	0	0	7.5078	7.4329	7.4704	7.4704	7.4704	94.56
	2	0	1	1	1	7.5780	7.5008	7.5394	7.5394	7.5394	95.44
-	3	0	2	2	2	7.3972	7.3929	7.3950	7.3950	7.3950	93.61
cul	4	1	0	1	2	7.5846	7.5058	7.5452	7.5452	7.5452	95.51
Lim	5	1	1	2	0	7.5279	7.4515	7.4897	7.4897	7.4897	94.81
bei	6	1	2	0	1	7.7387	7.8218	7.7803	7.7803	7.7803	98.48
Ě	7	2	0	2	1	7.3886	7.3894	7.3890	7.3890	7.3890	93.53
	8	2	1	0	2	7.5452	7.5410	7.5431	7.5431	7.5431	95.48
	9	2	2	1	0	7.4760	7.5262	7.5011	7.5011	7.5011	94.95
									Average	7.5170	95.15
									Max	7.7803	98.48
									Min	7.3890	93.53

Table 3: ANOVA for the sintered part at  $\alpha = 0.005$ 

Variable	Degree of freedom, $f_n$	Sum squared, S <sub>n</sub>	Variance, v <sub>n</sub>	Pure Sum squared, S <sub>n</sub> '	Variane e ratio, F <sub>n</sub>	Critical F value	Contribution , P <sub>n</sub>
A	2	0.140	0.070045	0.139	114.39	F0.005 7 77=6.4885	31.60
В	2	0.050	0.025065	0.049	40.94	F0.005 2 27=6.4885	11.13
С	2	0.183	0.091317	0.181	149.13	F0.005.2.27=6.4885	41.29
D	2	0.050	0.025001	0.049	40.83	F0.005 2 27=6.4885	11.10
error	27	0.017	0.000612			0.0007. 41 - 1	4.88
Total	35	0.439					100

Based on the ANOVA, the main effect of the experiments is calculated based on the highest average values as shown in Figure 1. As shown by the response plot in Figure 1, a combination of  $A_1$ ,  $B_2$ ,  $C_0$ , and  $D_1$  is the highest yield, i.e., sintering temperature 1360 °C, dwell time 240 minutes, heating rate 6 °C/minutes and cooling rate 8 °C/minutes. On the other hand, faster heating rate (20 °C/minutes) for sintering temperature at 1250 °C and dwell time of 90 minutes has been reported as the optimum sintering parameter by Ji et al. (2001).

The ANOVA shown in Table 3 signify that the effects of the variables are all significant at 99.5 % significance level. Hence the expected result at optimum performance is as shown in Table 4. The expected optimum performance is as high as 98.48 % of theoretical density while the range of the optimum performance based on 90 % confidence level is  $98.22 < \mu < 98.75 \%$  of the theoretical density. The optimum

parameter has been proven in the confirmation experiment that is conducted at the combined setting of  $A_1$ ,  $B_2$ ,  $C_0$ , and  $D_1$  and the result fell within the predicted 90 % confidence interval as shown in Table 4. A density at optimum performance reported by Ji et al. (2001) is 7.592 g/cm<sup>3</sup>, which is lower than that achieved by this study as shown in Table 4.



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Table 4: Optimum sintering parameter, optimum performance and confirmation experiment.

## Optimum parameter:

A1 B2 C0 D1

(Sintering Temperature, 1360 °C; dwell time, 240 minute; Heating rate, 6 °C/minute; Cooling rate, 8 °C/minute)

Optimum performance: 7.7803 g/cm3 or 98.48 % theoretical density

Confident interval:  $\pm 0.02$  at 90 % confident level ( $\alpha = 0.1$ )

Range of optimum performance : 7.7592 g/cm<sup>3</sup> <  $\mu$  < 7.8013 g/cm<sup>3</sup> or 98.22 % theoretical density <  $\mu$  < 98.75 % theoretical density

Confirmation experiment					
Repeat	1	2	3	4	Average
g/cm <sup>3</sup>	7.8377	7.8365	7.7296	7.7296	7.7834
% theoretical	1 12 12 1 2 1 2 1 2 1 2 1 2 1 2 1 2 1 2	selective states.	nana ar		1700 - 1700 - 1700 - 1700 - 1700 - 1700 - 1700 - 1700 - 1700 - 1700 - 1700 - 1700 - 1700 - 1700 - 1700 - 1700 -
density	99.21	99.20	97.84	97.84	98.52

#### 5. CONCLUSIONS

Sintered density of the water atomised SS316L MIM parts was optimised by using the Taguchi method. An  $L_9$  orthogonal array was used to vary the experiment variables. ANOVA showed that all the four sintering variables: sintering temperature, dwell time, heating rate and cooling rate, affected the sintered density significantly. The optimum sintering parameter were found to be A<sub>1</sub>, B<sub>2</sub>, C<sub>0</sub>, and D<sub>1</sub>, corresponding to sintering temperature of 1360 °C, dwell time of 240 minute, heating rate of 6 °C/minute and cooling rate of 8 °C/minute. Confirmation experiments indicated that when sintering SS316L at the optimum condition, a high 98.52 % of theoretical density can be achieved.

#### REFERENCES

Fu, G., Loh, N.H., Tor, S.B., Tay, B.Y., Murakoshi, Y. and Maeda, R., Injection molding, debinding and sintering of 316L stainless steel microstructures, *Applied Physics A: Materials Science and Processing*, Vol. 81, pp. 495-500, 2005.

Berginc, B., Kampuš, Z. and Šuštaršič, B., The influence of MIM and sintering process parameters on the mechanical properties of 316L SS, *Materiali in Tehnologije*, Vol. 40, No. 5, pp. 193-198, 2006.

Koseski, R.P., Suri, P., Earhardt, N.B., German, R.M. and Kwon, Y.S., Microstructure evolution of injection moulded gas and water atomized 316L stainless steel powder during sintering, *Materials Science and Engineering*, Vol. A390, pp. 171-177, 2005.

Li, S., Huang, B., Li, D., Li, Y. Liang, S. and Zhou, H., Influence of sintering atmosphere on densification process of injection moulded gas atomised 316L stainless steel, *Powder Metallurgy*, Vol. 46, No. 3, pp. 241-245, 2003.

Suri, P., Koseski, R.P. and German, R.M., Microstructural evolution of injection molded gas and water atomized 316L stainless steel powder during sintering, *Materials Science and Engineering*, Vol. A402, pp. 341-348, 2005.

Khairur Rijal Jamaludin, Norhamidi Muhamad, Mohd Nizam Ab. Rahman, Sri Yulis M. Amin and Murtadhahadi, Molding parameter optimization for reducing a green defects for Metal Injection Moulding green part, (in Malay) *The Journal of The Institution of Engineers, Malaysia*, Vol. 69, No. 2, pp 40-46, 2008a.

Khairur Rijal Jamaludin, Norhamidi Muhamad, Mohd Nizam Ab. Rahman, Sri Yulis M. Amin and Murtadhahadi, Analysis of Variance on the Metal Injection molding parameters using a bimodal particle size distribution feedstock, *Proceeding of International Conference of Mechanical and Manufacturing Engineering (ICME)*, pp 1-5, Johor Bahru, Malaysia, 2008.

Jamaludin, K.R., Muhamad, N., Amin, S.Y.M., Ab. Rahman, M.N., Ismail, M.H. and Murtadhahadi, I., Injection Molding Parameter Optimization Using Taguchi Method For Highest Green Strength For Bimodal Powder Mixture with SS316L in PEG, and PMMA, Advances in Powder Metallurgy & Particulate Materials, Vol.1, pp. 186-194, 2008.

Ji, C.H., Loh, N.H., Khor, K.A. and Tor, S.B, Sintering study of 316L stainless steel Metal Injection Molding parts using Taguchi method: final density, *Materials Science and Engineering*, Vol. A311, pp. 74-82, 2001. Park, S.H, Robust design and analysis for quality engineering, UK: Chapman & Hall, 1996.

Kang, S.J.L, Sintering densification, grain growth and microstructure, UK: Elsevier Butterworth – Heinemann, 2005.

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