Optimising sintering parameters on the characteristic of injection moulded 316L stainless steel utilising water soluble system

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ABSTRACT

This paper describes an optimising sintering process in metal injection molding (MIM) of 316L SS employing a binder system consisting of palm stearin (PS), stearic acid (SA), polyethylene (PE) and thermoplastic rice starch (TPRS). The Design of Experiment (DOE) for this study used the Taguchi method with orthogonal array L_9 (3^3) for optimization of sintered parts. The outcomes were observed in achieving the maximum sintered density yield. The part was injection moulded with a pressure of 7 bars, an injection temperature of 160°C, an injection time of 10 seconds and a holding time of 25 seconds. Subsequently, the part was solvent debinded by immersing in n-heptane at 60°C for 6 hours and thermal debinding was performed at 450°C with a heating rate of 1°C/min and one-hour soaking time. Sintering parameters optimized were sintering temperature ranging from 1300°C to 1380°C, heating rate range of 1°C/min to 5°C/min and holding time range of 45 minutes to 90 minutes. The optimum sintered density was achieved with a sintering temperature of 1340°C, a heating rate of 3°C/min and a holding time of 60 minutes. Analysis of variance (ANOVA) revealed that the heating rate (60.29%) was the most influential parameter that contributed to sintered density followed by holding time (32.87%) and sintering temperature (6.84%).

Keywords: *Metal Injection Molding; Taguchi Method; Stainless Steel; Sintering.*

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Introduction

Several advantages in production technologies are achievable through Metal Injection Molding (MIM). The MIM is a manufacturing process comprising of four processing steps namely, mixing, molding, debinding and sintering. In the mixing process, the metal powder particles are mixed with a binder to form a feedstock for molding. Molding is formed by shaping the parts from feedstock using an injection molding machine. The debinding process removed the binder in the green parts in a sequence of steps [1]. Once the binder is removed, the parts will be sintered to obtain the required mechanical properties. Some unique advantages of MIM are producing parts with complex geometries, simplifying the production process and reducing manufacturing costs. According to Escobar et al. [2], 316L SS is one of the most widely used materials due to its corrosion resistance, excellent mechanical strength and good biocompatibility properties.

Sintering process is one of the highly critical factors that can influence mechanical, chemical and dimensional properties of the MIM parts [3]. A previous study by Ye et al. [4] demonstrated the effects on specimens by sintering temperature and sintering atmosphere. The study concluded that oxidation during sintering can be avoided by using vacuum or argon atmosphere and the amount of weight gain increased with increasing sintering temperature. A research by Ahmad et al. [3] indicated the influence of sintering temperature and cooling rate on mechanical properties of specimens. Low sintered density obtained when low cooling rate was used due to the presence of porosity. The strength of specimen also improved when high cooling rate was used even at high sintering temperature.

According to Raza et al. [5], some specimens were sintered in different atmospheric conditions and maximum densification was achieved when the parts were sintered in vacuum and hydrogen atmosphere. When the parts were sintered in nitrogen, high tensile strength was achieved. Wahab et al. [6] conducted research on the potential of starch for medical device applications. The sintering process was conducted at different temperatures in a vacuum environment. The part was soaked for 1 hour with a heating rate of 3°C/min in order to prevent crack formation. Past research by Omar et al. [7] indicated that the percentage of specimen's shrinkage increased when sintering temperature was increased. Increased in sintering temperature led to compact densification, thus increasing the sintered density.

In this study, the influence of sintering parameters on characteristics of injection moulded 316L SS using water soluble system was studied. The sintering parameters optimized were sintering temperature, heating rate and holding time. The Taguchi method was used to conduct a design of experiments (DOE) for optimization of sintering parameters in order to obtain the highest density for the sintered part. DOE is a statistical technique

to study the effects of multiple variables simultaneously and determined the best factor combination. According to Norhamidi et al. [8], the design of experiment (DOE) is the best option when facing situation that depends on many influencing factors or parameters. The Taguchi method is used to analyse the optimal performance characteristics via a set of factors. The Taguchi method has gained interest among researchers because it provides effective techniques in finding the best parameters which can minimize costs and defects in an experiment. It is also used for the purpose of designing and improving the quality of the product produced [9].

Jamaludin et al. [10] conducted a research of sintering parameter optimisation for the final density. The optimum parameters were found to be 1360°C of sintering temperature, dwelling time of 240 minutes, heating rate of 6°C/min and cooling rate of 8°C/min. Theoretical density of 98.52% was achieved by using optimum condition of sintering. Previous research by Mohamad Nor et al. [11] showed a sintering study of MIM using the Taguchi method. Optimized parameters used were sintering temperature of 1300°C, sintering time of 120 minutes, heating rate of 4°C/min and cooling rate of 9°C/min. The results showed sintering temperature was the most influential parameter that contributed to highest sintered density. The signal-to-noise (S/N) ratio and analysis of variance (ANOVA) were applied to study the optimum levels and contribution of each parameter to sintered parts.

Methodology

Characterization of binder components

The binder components comprised of PS, SA, PE and TPRS. The density of each binder components was measured to calculate feedstock preparation in the MIM process by using the Gas Pycnometer machine. The Differential Scanning Calorimeter (DSC) machine was used in determining the melting point which may aid to decide the temperature for the mixing process and ensure that all components will melt and form a homogeneous mixture.

Samples preparation

The calculation of feedstock involving important parameters such as density and melting point of each binder components were carried out. For the preparation of feedstock, a mass of 316L SS metal powder and binder components comprising TPRS, PS, PE and SA were determined for the mixing process. Prior to mixing, the TPRS was prepared by mixing RS with PEG in a beaker by using a water bath with a temperature of 60°C for 15 minutes. The mixing process was conducted by using the Z-blade machine. Parameters used were 150°C heating temperature, 50 rev/min speed and

mixing time of 2 hours. Table 1 shows the powder loading and binder composition used in this experiment.

Table 1: Composition of powder and binder components

Powder loading	Binder composition
63 : 37	TPRS : 35 PS : 35 PE : 25 SA : 5

Parameters of injection molding was referred to Azizul Ghafar [12] as the researcher used these set of parameters to obtain the highest strength of the green part. Optimized parameters of injection molding were injection pressure of 7 bar, injection temperature of 160°C, injection time of 10 seconds and holding time of 25 seconds. Injection molding was performed using a vertical injection molding machine.

The debinding parameters used were based on research findings by Wahab et al. [6]. The green parts were immersed in n-heptane at 60°C for 6 hours. After the solvent debinding process, the brown parts were left to dry in the oven for 12 hours at 60°C. Thermal debinding was performed at 450°C with a heating rate of 1°C/min and one-hour of soaking time.

Sintering process was performed along with thermal debinding in a High Temperature Tube Furnace by using argon gas as sintering atmosphere. The brown parts were sintered at different parameters based on the arrangement laid out in Table 2.

Design of Experiment (DOE)

DOE was utilized in this research during the sintering process. Based on parameters selected, L_9 (3^3) orthogonal array was used for this experiment which involved nine experiment trials with three parameters and three levels of factor. The three parameters involved were sintering temperature, heating rate and heating time. Table 2 shows the parameters involved and the arrangement of parameters is tabulated in Table 3.

Table 2: DOE of sintering process

Process parameters	Symbol	Level		
		1	**2**	**3**
Sintering temperature (°C)	A	1300	1340	1380
Heating rate (°C/min)	B	1	3	5
Holding time (min)	C	45	60	90

Table 3: Orthogonal array $\mathbf{L_9}$

Experiment	Sintering temperature (°C)	Heating rate (°C/min)	Holding time (min)
1	1300	1	45
2	1300	3	90
3	1300	5	60
4	1340	1	90
5	1340	3	60
6	1340	5	45
7	1380	1	60
8	1380	3	45
9	1380	5	90

Dimension, density, hardness, strength and microstructure were determined in order to characterize the sintered parts of 316L SS. The dimension of the sintered part was measured by using a Vernier caliper as shown in Figure 1. The density was measured using the Archimedes method and calculated as a percentage of the theoretical value (MPIF standard 10). The hardness test was conducted by using Hardness Rockwell A (MPIF standard 43) whilst the tensile test (MPIF standard 10) was performed to determine the strength of the sample by measuring the force required to break the sample while Scanning Electron Microscope (SEM) was used to analyse the morphology of the sintered part.

Results and Discussion

Characterization of materials

The gas atomised 316L SS powder used in this study was obtained from Sandvik Osprey with an average particle size of 22 microns. As shown in Figure 1, all the particles were approximately spherical in shape with some particles relatively larger in size, while some others were very small.

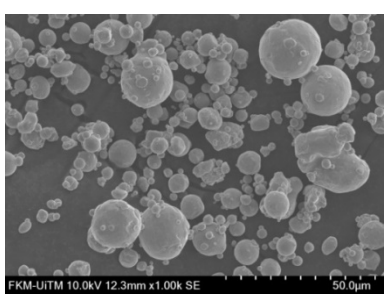


Figure 1: Scanning electron micrograph (SEM) of 316L SS powder

Binder components were characterized to determine the density and melting point. The graph of melting point with heat flow versus temperature was plotted as shown in Figure 2. The peak of the graph represents the melting point of each binder components [13] and the value is tabulated in Table 4. The density of the metal powder 316L SS with its binder components is also tabulated in Table 4.

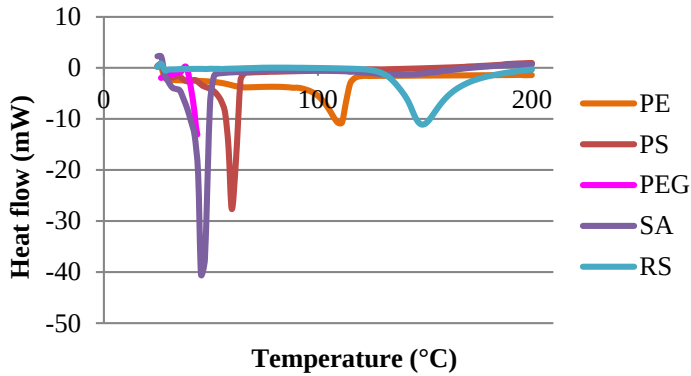


Figure 2: DSC analysis of binder components

Table 4: Density and melting point of powder 316L SS and binder components

Type of powder/binders	Density (g/cm^3)	Melting point (°C)
316L SS	7.99	
PE	0.93	110.35
PS	0.97	60.17
PEG	1.23	46.57
SA	1.01	46.08
RS	1.50	148.78

Analysis of injection moulded part

Figure 3 shows the micrograph of fracture surface of the injection moulded part which was observed using a Scanning Electron Microscope (SEM). From the micrograph observed, it can be seen that the binder components comprised of PE, PS, SA and TPRS which filled up the interstitial sites of the 316L SS particles. The binder acted as a backbone to support the part until the debinding process and build a binder network to hold the particles in shape. This observation is in good agreement with the research findings by Wahab et al. [6].

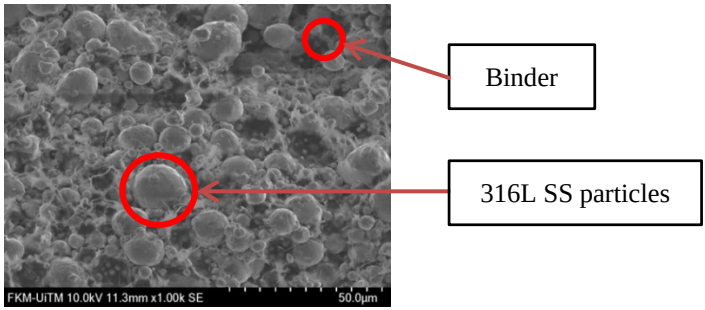


Figure 3: SEM of green fractured surface

Analysis of sintered part

Table 5 shows the micrograph of fracture surface of the sintered parts observed under SEM. Sintering parameters used for sintered parts were sintering temperature ranging from 1300°C to 1380°C, heating rate of 1°C/min to 5°C/min and holding time of 45 minutes to 90 minutes. Each of the sample has different pore sizes and pore distributions based on sintering temperature, heating rate and holding time which were used as sintering parameters. It can be observed that the sintered parts exhibited ductile surface failure regardless of parameters used for this experiment. Spherical dimples can be observed on the fracture surface. This type of failure is also called a cup-and-cone fracture because one of the mating surfaces is in the form of a cup while the other one is like a cone. As shown in Table 5, most of the fracture surface has a cone shape due to shear fracture which occurred at 45° angle relative to the tensile direction [19].

It can be seen that as the temperature increases, there was less porosity observed. The geometry of irregular shaped pores become larger and more rounded when sintering temperature increased. The results obtained is similar to a study conducted by Ji et al. [14] which showed that the number of pores decreases and size of the pores become larger when there was an increase in temperature during the sintering process. From the micrograph, increased in heating rate resulted in less porosity and finer grains. According to Omar et al. [7], a too low heating rate will cause nearly no densification while a too high heating rate will result in warpage and distortion of sintered parts. As heating rate increased from 1°C/min to 5°C/min, complete surface diffusion took place that caused the number of pores to reduce. Research conducted by Coovattanachai et al. [15] showed that a slow heating rate caused coarser grains due to a longer time of atomic diffusion. Increased in holding time produced a refined microstructure [16]. However, the pore size, pore distribution and shape of grain depended on a combination of sintering parameters utilized in this experiment.

Table 5: SEM of sintered parts according to L_9 orthogonal array

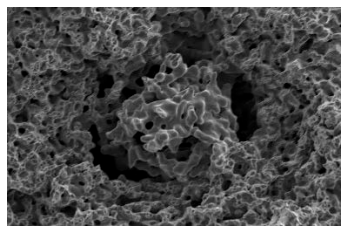
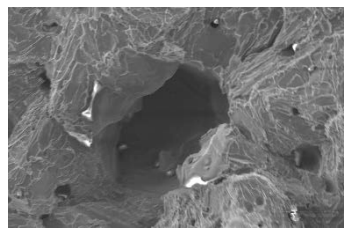
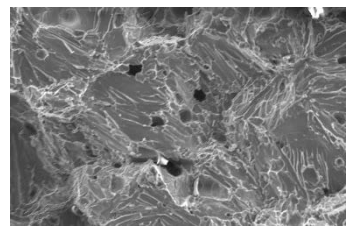
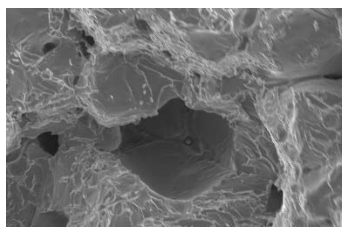
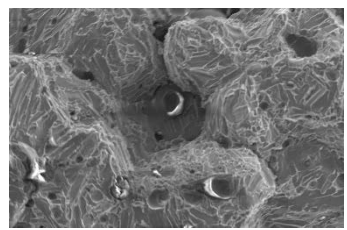
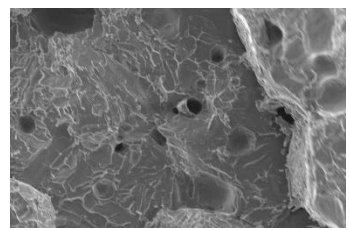
Holding time / **Sintering Temp.**	**45 minutes**	**60 minutes**	**90 minutes**
1300°C	 1°C/min	 5°C/min	 3°C/min
1340°C	 5°C/min	 3°C/min	 1°C/min

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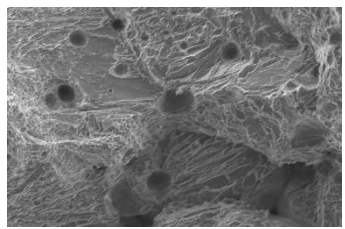
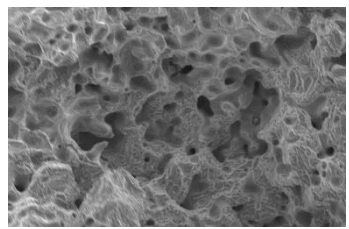
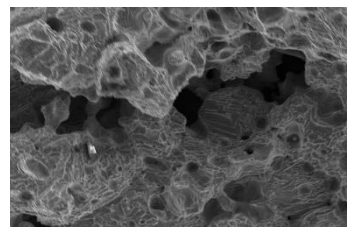
Table 6: SEM of sintered parts according to L_9 orthogonal array (cont.)

Holding time / **Sintering Temp.**	**45 minutes**	**60 minutes**	**90 minutes**
1380°C	 3°C/min	 1°C/min	 5°C/min

SEM of fracture surface for sintered part having optimum density (Figure 4) shows the type of failure which is known as cup-and-cone fracture because one of the mating surfaces is in the form of a cup while the other one is like a cone. Most of the fracture surface has cone shape due to shear fracture which occur at 45° angle relative to the tensile direction.

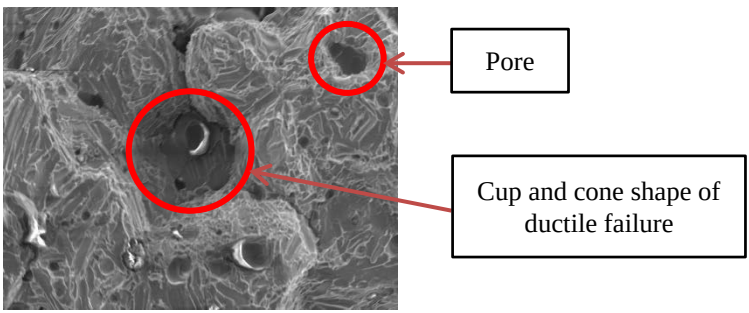


Figure 4: SEM of sintered fracture surface using a combination sintering temperature of 1340°C, 3°C/min of heating rate and 60 minutes holding time

For each experiment, the test was conducted on three samples and the mean value for the experiment was recorded. The shrinkage percentage showed that the length and weight of a sintered part are smaller compared to a green injection moulded part. Figure 5 shows the dimension of a green injection moulded part and a sintered part. Tensile strength and hardness tests were conducted on the sintered part. The results of physical and mechanical properties of the sintered part with different parameters are presented in Table 6.

Figure 5: Dimensional comparison between a) green injection moulded part and b) sintered part

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Table 7: Physical and mechanical properties of 316L SS sintered parts

Experiment	Shrinkage (%)		Tensile strength (MPa)	Hardness (HRA)
	Length	Weight		
1	8.77	6.88	257.99	48.90
2	10.75	7.13	298.78	48.67
3	10.21	6.96	283.17	49.60
4	10.72	6.98	201.08	48.20
5	12.26	6.87	338.18	50.00
6	11.62	7.08	296.51	48.17
7	11.45	7.05	391.39	45.73
8	9.10	6.80	242.20	48.33
9	10.69	7.04	307.57	47.27

Analysis of S/N Ratio

S/N ratio has been utilized as the Taguchi technique to measure the quality characteristic which deviating from the desired value [8]. By using S/N ratio, larger-the-better quality characteristic was used in this experiment to observed the highest density of sintered part. S/N ratio was calculated by using Equation 1 in order to obtained the highest value of signal to noise ratio. Value 'y' from Equation (1) was taken from density value as shown in Table 7 while 'n' value is the mean number of experiment.

$$\text{S/N} = (-10)x\ log\ \sum \frac{\frac{1}{(y^2)}}{n} \quad (1)$$

The calculated S/N ratio for each of experiment is shown in Table 7 and graph of S/N ratio for each of sintered parameter was plotted in Figure 6. Based on the graph, it can be seen that each parameter has the highest S/N ratio and the best combination of factors are A2, B2 and C2 which are corresponding to the sintering temperature of 1340°C, heating rate of 3°C/min and 60 minutes holding time. These parameters are at optimum level which resulting in highest density of sintered part. From the graph plotted, the highest S/N value for each parameter used in this experiment is tabulated in Table 8.

Table 8: S/N ratio of density

Experiment	Parameters			Density			SNR
	A	B	C	1	2	3	
1	1	1	1	6.25	7.49	5.53	15.96
2	1	2	3	7.35	7.41	6.68	17.05
3	1	3	2	7.41	7.42	6.10	16.76
4	2	1	3	6.73	7.09	7.34	16.95
5	2	2	2	7.42	7.43	7.03	17.25
6	2	3	1	6.39	7.33	6.03	16.28
7	3	1	2	6.8	6.84	7.29	16.89
8	3	2	1	7.09	7.13	7.20	17.08
9	3	3	3	7.18	5.75	6.88	16.27

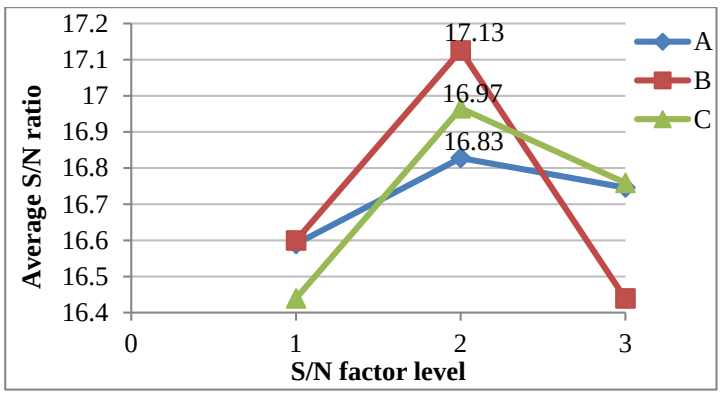

Figure 6: Response plot for S/N ratio of sintering temperature, heating rate and holding time

Table 9: Optimize sintering parameters

Parameters	Highest S/N value	Selected S/N value	Selected parameters
Sintering temperature (°C)	16.83	A2	1340
Heating rate (°C/min)	17.13	B2	3
Holding time (min)	16.97	C2	60

Analysis of Variance (ANOVA)

ANOVA has been employed to demonstrates the relative significant of parameters and contributions of each parameter in an experiment [17]. Table 9 shows the result of ANOVA before pooling in which to determine the most significant parameter for this experiment. Heating rate was the most influence parameter which affect the characteristic of sintered 316L SS part. Heating rate represents the most contribution which is 60.29% while holding time contribute 32.87% for the sintered part. The lowest contribution is represent by sintering temperature which showed the lowest value, 6.84%.

Table 10: ANOVA before pooling

Factor	Parameter	DOF	Sn	Variance, Vn	F	% Contribution
A	Sintering temperature (°C)	2	0.08	0.04		6.84
B	Heating rate (°C/min)	2	0.77	0.39		60.29
C	Holding time (min)	2	0.42	0.21		32.87
Error		0				
Total		6	1.28	0.64		100.00

The percentage contribution of heating rate is reduced to 27.42% after pooling as shown in Table 10. Two parameters were pooled in order to get F value correspond to F-table. F value should be compared with the standard table value which is called F-table for the desired confidence level [18]. Despite pooling of two parameters, F value does not exceeds the standard value in F-table. In this case where the F-test cannot be performed, pooling should be conducted by considering the pooling parameters starting with a lower percentage influence. There is a rule where pooling is done when Sum of Squares, Sn value of the influence factor is 10% or lower than the most influential factor [18]. For this case, the rule of pooling could not be applied to the ANOVA. Eventhough the F-test could not be conducted, pooling should be continued until the error degree of freedom (DOF) approximately half the total of DOF of the experiment. Two parameters were pooled which begin with holding time because it has the lowest percentage influence followed by sintering temperature. When two parameters were pooled, value of error DOF is 4 which exceeds half the total DOF of the experiment. It is important to have error DOF value in order to calculate the confidence interval for this experiment.

Table 11: ANOVA after pooling

Factor	Parameter	DOF	Sn	Variance, Vn	F	% Contribution
A	Sintering temperature (°C)	POOLED				
B	Heating rate (°C/min)	2	0.77	0.39	1.52	27.42
C	Holding time (min)	POOLED				
Error		4	0.51			72.58
Total		6	1.28	0.25		100.00

Confidence Intervals (C.I)

The confidence intervals (C.I) is always calculated at a confidence level. For result confirmation of an experiment, multiple samples were tested to obtained the expected result which is expressed as an estimate of the mean of the average performance. C.I. shows the optimum sintering parameter, optimum performance and confirmation experiment.

Table 12: Optimum sintering parameter, optimum performance and confirmation experiment

Significance optimum parameter : B2					
Optimum performance calculation : $\overline{\overline{T}} + (\overline{B2} - \overline{\overline{T}})$ **16.72 + (17.24 - 16.72) = 17.24 dB**					
Average performance			: 16.72		
Confident interval at 90% confidence level ($\alpha = 0.1$)			: ± 0.80		
Expected result at optimum performance, μ			: $16.44 < \mu < 18.04$		
Confirmation Experiments					
Experiments	**1**	**2**	**3**	**Average**	**S/N ratio**
Selected value	6.96	7.07	7.23	7.09	17.01

$$\text{C. I.} = \pm\left[\frac{F(1,n_2) \times V_e}{N_e}\right]^{0.5} \quad (2)$$

Equation (2) is formula used to obtained the C. I. value, where $F(1,n_2)$ is the F value from F-table, n_2 is the DOF for the error term, V_e is the variance of the error term (from ANOVA) and N_e is the effective number of replications. The expected optimum performance is in the range of the

optimum performance based on 90% confidence level which is $16.44 < \mu < 18.04$. The optimum parameter was proven in the confirmation experiment that was conducted with the combination parameters of A2, B2 and C2. The combination parameters of A2, B2 and C2 is the optimize parameter in obtaining highest sintered density of sintered part. The confirmation experiment performed is based on highest S/N ratio of each parameter. The result fell within the predicted 90% confidence interval as shown in Table 11. The optimum density can be achieved up to 7.09 g/cm^3.

Conclusion

By using the Taguchi method, the sintered density of injection moulded 316L SS was optimized. An L_9 orthogonal array was used to accommodate the 3^3 experiment. ANOVA showed that all three sintering parameters which were sintering temperature, heating rate and holding time affected the sintered density significantly. The optimum sintering parameters were found to be A2, B2 and C2 corresponding to sintering temperature of 1340°C, heating rate of 3°C/minute and holding time of 60 minutes. The confirmation experiments indicated that when sintering 316L SS at the optimal condition, a high 92.27% theoretical density was achieved. Future work should focus on optimisation of other sintering parameters such as sintering environment and cooling rate as well as increasing the number of parameters level in order to achieve precise result.

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