THE IMPACT OF SINTERING PROCESS PARAMETERS ON DENSITY OF PARTS PRODUCED BY METAL INJECTION MOULDING (MIM)

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ABSTRACT

The identification of significant process parameters during sintering of brown parts in Metal Injection Moulding (MIM) is essential because improper control of these parameters can result poor parts. The controlled parameters used for optimization in this work include sintering temperature, heating rate, sintering time, cooling rate and sintering atmosphere. These parameters had been optimized using analysis of variance (ANOVA) for signal to noise ratios obtained in experiment performed by following Taguchi L_{16} orthogonal array. The ANOVA was used to obtain the contribution of significant process parameters for final density of parts. Results showed that four out of the five factors were the significant factors. The sintering temperature had a contribution of 14.37% at a confidence level of 95%, heating rate had a contribution of 14.65% at a confidence level of 95%, sintering time had a contribution of 59.58% at a confidence level of 99%, cooling rate had a contribution of 10.70% with a confidence level of 95%, and sintering atmosphere was observed to be insignificant factor for final density attained by the specimen.

KEYWORDS: Analysis of Variance (ANOVA); Orthogonal Array; Metal Injection Moulding (MIM); Sintering

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INTRODUCTION

Metal Injection Moulding (MIM) is one of the promising technologies to process metal powders into parts of desired shapes. The MIM process incorporates the traditional shape-making capability of plastic injection moulding and materials flexibility of powder metallurgy [1]. The process consists of four main steps: mixing, injection moulding, debinding and sintering. In the mixing step, the powder is mixed with a binder to form a homogeneous feedstock. The binder is key component, which caters the necessary flowability and formability for moulding [2]. During injection moulding a green part with the desired shape is formed by the feedstock flow into a mold under pressure. After moulding, the binder holds the particles in place. The binder is then withdrawn in the debinding step and the debound brown part is sintered to achieve the required mechanical properties. The geometrical accuracy and mechanical properties of the final parts after sintering depend strongly on the process parameters in the different stages [3,4]. The classical design of experiment (DOE) technique has been used by many authors [5-7] for the sake of optimization of single process parameters at a time. In order to obtain high efficiency in the planning and analysis of experimental data, the Taguchi method is recognized as a systematic approach for design and analysis of experiments to improve the product quality. This method has been applied by many authors to investigate and optimize the process parameters [8-13]. The success of MIM process is highly dependent on sintering process [16-21]. The main objective of this paper is to optimize the sintering process parameters to attain high density of final parts.

MATERIALS AND METHODS

To make the working material, the SS316L stainless steel powder was mixed with the binder comprised of polyethylene glycol (PEG), polymethyl methacrylate (PMMA), paraffin wax and stearic acid (SA) to form the feedstock for moulding. The main advantage and utility of using PMMA/PEG binder is that it can be removed from the mouldings in a comparatively short time. The SS316L metal powder used in this research was supplied by Osprey®. The chemical composition of the steel powder used is presented in Table 1. The particle size distribution of metal powder is given in Table 2. The percentage concentration of constituents by weight and densities are presented in Table 3. The details of the binder ingredients are given in Table 4.

Table 1: Composition of SS316 L Powder (Report given by Osprey® along with the Powder)

Element %								
C	Si	Mn	P	S	Cr	Ni	Mo	Fe
0.018	0.55	1.5	0.031	0.017	16.9	11.6	2.2	balance

Table 2: Size Distribution of SS316 L Powder (Report given by Osprey®)

Powder Tests Report by Sandvik Osprey Ltd.				
d10	d50	d90	-53 µm	Tap Density
3.9 µm	13.0 µm	36.6 µm	99.2 %	5.0 gm/cc

Table 3: Theoretical Density of Constituents of SS316 L Powder

Element	Percentage Concentration	Theoretical Density
C	0.018	2.267
Si	0.55	2.33
Mn	1.5	7.47
P	0.031	1.823
S	0.017	1.96
Cr	16.9	7.14
Ni	11.6	8.9
Mo	2.2	10.28
Fe	67.184	7.874
SS 316 L	100	7.88146

Table 4: The Binder Ingredients

Designation	Manufacturer	Amount (%)	Melting point °C	Boiling point °C	Density (gm/cc)
Polymethyl Methacrylate (PMMA)	Vetec	65	157.77	200	1.19
Polyethylene Glycol (PEG)	Rankem	08	35-40	250	1.22
Paraffin Wax	Thermo Fischer Scientific	25	60-62	370	0.90
Stearic acid (SA)	Thermo Fischer Scientific	02	70.1	383	0.94

Feedstock Formulation

Both the metal powder and binder were mixed thoroughly for 90 minutes with the help of a Brookfield Rheometer in the desired proportion under precise weight and temperature control condition. The calculated amount of metal powder, PMMA, PEG, paraffin wax and stearic acid were weighed and mixed together. The mixing was carried out at 160°C and 40 rpm to attain a homogeneous distribution of the powder particles and binder in feedstock. After thorough mixing, the mixture was first dried in air at ambient temperature for 2 hours and then in an oven at a temperature of 50°C for 1 hour. After compounding the feedstock was allowed to cool to at ambient temperature and then granulated in a rotary feedstock granulator. The SEM micrograph of SS316L powder used for study is shown in Figure 1.

Production of Test Specimen

A four-cavity mould was specifically designed and made with cavities in accordance with MPIF Standard 50 and ASTM Standard E8-98. The impact specimen geometry needed for this study was a 5mm x 10mm x 55mm unnotched bar. Using the known shrinkage factor for the given feedstock, the impact bars were molded to produce oversize green parts as shown in Figure 2. The subsequent processing produced parts with the final dimensions as specified in MPIF Standard 59 [14]. Either no machining or very fine machining was needed to prepare the Charpy impact bars for testing. The green parts and sintered parts are shown in Figure 2 and Figure 3 respectively. The final parts shown in Figure 4 were used to find the final density.

Figure 1: SEM Micrographs of SS316L Powder used for Study.

Figure 2: MPIF 59 Based Impact Test Bar (Specimen Size Before Sintering).

Figure 3: The Samples Produced by Injection Moulding.

Figure 4: The Samples Produced After Sintering.

Injection Moulding Procedure

Injection moulding process comprises of heating of the feedstock material to binder melting temperature, forcing the molten material into the mould cavities, holding at high pressure, then cooling and ejection of the molded parts out of the mould cavity. In the experimental work, a Demag injection moulding machine with microprocessor control was used. On the machine, the injection pressure, injection temperature, mould temperature, holding pressure, injection speed, holding time and cooling time were set at the desired values. Since, the powder loading is an external factor; it is not to be taken care by the machine control. Each set of values was repeated five times to make samples at each processing conditions after the machine has come to smooth functioning. All the test parts were produced by using only virgin feedstock.

Debinding Procedure

The green parts were debound through the solvent and thermal debinding techniques to remove the binders effectively. In the first step, solvent extraction was used to extract out the PEG and paraffin wax from the green parts. The green specimens were immersed in high temperature distilled water with continuous stirring. The leached specimens were then dried in an oven to remove the remains of water completely and then cooled it again. The second step, referred to as thermal debinding was used to remove the PMMA and stearic acid after solvent debinding. The leached specimens were put into an alumina tray in which the surrounding space was filled with alumina powder to avoid any distortion of the specimens. The thermal debinding was achieved in a vacuum furnace. The brown part was allowed for a slow cooling to ambient temperature (27°C) to release the residual stress from the part.

Sintering Procedure

The objective of this experiment was to find the significant factors in sintering process, their contribution and optimised levels. As good quality parts with desired mechanical properties can be ensured only after proper sintering, an analysis of variance (ANOVA) is utilized to identify the significant level of each variable.

If full factorial design is used in all the four factors with four levels then it will develop 256 combinations. But, the modified L_{16} can be used to accommodate four factors with four levels is based on the concept for conversion, which in turn dependent upon the concept of degrees of freedom [8]. It is notable that a two level OA has one piece of information associated with it: the comparison of the average of all the first level tests compared to the averages of all of the second level tests. While, a four level factor has three pieces of information concerning with it: the first- to second level effect, the second to third level effect, and the third to fourth level effect [22]. Using the investigations [11, 20] and the expertise of the sintering process five main parameters were considered to study. The processing variables and their values at different levels are shown in Table 5.

The experimental results were converted in S/N values for optimization of parameters. The S/N ratio (higher the better) is used for analysis. To find the confidence level and variance of the data ANOVA is used for effective output. The confidence level is measured from the variance of each parameter. In order to verify a confirmation test was conducted within the range of optimum performance calculation.

Table 5: Controllable Process Parameters for Sintering of Specimens

Process Parameters	Symbol	Level 1	Level 2	Level 3	Level 4
Sintering temperature (°C)	T_s	1260	1300	1340	1380
Heating rate (°C/min)	T_h^*	4	8	12	16
Sintering time (minutes)	t_s	60	80	100	120
Cooling rate (°C/min)	T_c^*	5	10	15	20
Sintering atmosphere	p	Vacuum	N2	--	---

RESULTS

The calculated values of S/N ratio to access the variation in density due to variation in sintering parameters are shown in Table 6. The analysis of variance made by using S/N ratio to find the significant factors and their contribution is expressed in Table 7. From Table, it can be observed that four out of the five factors are the significant factors. Since, there is one insignificant factor, so pooling is required. The sintering temperature has a contribution of 14.37% at a confidence level of 95%, heating rate has a contribution of 14.65% at a confidence level of 95%, sintering time has a contribution of 59.58% at a confidence level of 99%, cooling rate has a contribution of 10.70% with a confidence level of 95%, and sintering atmosphere is observed to be insignificant factor for density attained by the specimen.

The optimum level of parameters can be obtained by selecting the highest values of S/N ratios from respective column in Table 6. The optimum level for Density occurs at $(T_s)_4(T_h^*)_3(t_s)_4(T_c^*)_2(p)_2$. From Table 6 it can also be noted from the rank of parameters that the variation in the S/N ratio value with the change in the value of the parameter is maximum for sintering time and minimum for sintering atmosphere.

Table 6: Response Table for Signal to Noise Ratios (Larger is Better)

Level	T_s	T_h^*	t_s	T_c^*	p
1	17.41	17.37	17.24	17.46	17.46
2	17.42	17.46	17.49	17.51	17.46
3	17.44	17.55	17.53	17.49	--
4	17.57	17.45	17.56	17.37	--
Delta	0.16	0.18	0.32	0.14	0.00
Rank	3	2	1	4	5

Table 7: Analysis of Variance using S/N Ratios for Density

Factors/Source	DOF, ν	Sums of squares	Variance, V_n	Variance Ratio, F_n	Significance Level, α	Pure Sum Square	Contribution, P in %
T_s	3	0.06378	0.02126	107.38	0.05	0.06318	14.37
T_h^*	3	0.06501	0.02167	109.45	0.05	0.06441	14.65
t_s	3	0.26249	0.08749	441.88	0.01	0.26189	59.58
T_c^*	3	0.04763	0.01588	80.19	0.05	0.04703	10.70
p	(1)	0.00007					
Residual Error	3	0.000594	0.000198				0.70
Total	15	0.43953					100

The required response the analysis of the trend of performance characteristics with respect to variation of the controllable factor are shown in Figure 5 and Table 6.

Figure 5: Main Effects Plot for Mean Values of Density due to Controlled Sintering Parameters.

Figure 6: Interaction Plot for Sintering Temperature (°C), Heating Rate (°C/min), and Sintering Time (Minutes).

Figure 7: Interaction Plot for Heating Rate (°C/min), Sintering Time (Minutes), and Cooling Rate (°C/min).

Figure 8: Interaction Plot for Sintering Temperature (°C), Sintering Time (Minutes), and Cooling Rate (°C/min).

Table 8: Factor Levels for Prediction

Sintering temperature (°C)	Heating rate (°C/min)	Sintering time (minutes)	Cooling rate (°C/min)	Sintering atmosphere
1380	12	120	10	Nitrogen

CONFIRMATION TEST

Since, the sintering temperature, heating rate, sintering time, and cooling rate are the significant factors; the optimum value of density will depend on all these factors and could be estimated by equation (1) at the optimum levels shown in Table 8.

$$\mu_p = \bar{T} + [(T_s)_4 - \bar{T}] + [(T_h)_3 - \bar{T}] + [(t_s)_4 - \bar{T}] + [(T_c)_2 - \bar{T}] \quad (1)$$

Hence, the expected density at optimum condition is:

$$\mu_p = 7.558 + 7.546 + 7.556 + 7.508 - (3 \times 7.4691) = 7.7607 \text{ gm cm}^{-3}$$

The 95% confidence interval (CI) for the expected yield from the confirmation experiment can be calculated using equation (2) as follows:

$$CI = \left(F_{\alpha}(\vartheta_1, \vartheta_2) V_e \left[\left(\frac{1}{n_{eff}} \right) + \left(\frac{1}{r} \right) \right] \right)^{1/2} \quad (2)$$

Where,

$$n_{eff} = (N / (1 + \text{total degree of freedom of all factors used for estimating } \mu))$$

r = sample size for the confirmation experiment, $r \neq 0$. $F_{\alpha}(\vartheta_1, \vartheta_2)$ is the variance ratio of ϑ_1 and ϑ_2 at level of significance α . The confidence level is $(1-\alpha)$, ϑ_1 is the degree of freedom of mean (equal to 1) and ϑ_2 is the degree of freedom for the pooled error. Variance for pooled error is V_e . The confidence interval indicates the maximum and minimum levels of the optimum performance.

$$CI = \{ 10.1 \times 0.000198 [(1/6.15) + (1/5)] \}^{1/2} = \pm 0.02692$$

Therefore, expected density at optimum condition = 7.4691 ± 0.02692

To confirm the prediction, another five samples were made at the recommended setting $(T_s)_4(T_{\square}^5)_3(t_s)_4(T_{\square}^5)_2$ and $(p)_2$ as shown in Table 8. The results are shown in Table 9.

Table 9: Results of Confirmation Experiments

Characteristic	Replication at Optimum Process Parameters					Average	Minitab predicted value
	R1	R2	R3	R4	R5		
Density (gm cm ⁻³)	7.4501	7.5232	7.5863	7.5291	7.6844	7.5546	7.7582

It is found that the average density obtained from the confirmation experiment is close to the mean of 95% confidence interval. From Table 9 it can also be noted that the experimental results are close to the predicted result by Minitab 17 software. The difference between measured and predicted values is about 2.69%. The deviation occurs because the Minitab software uses linear regression model for prediction.

CONCLUSIONS

Sintering temperature is observed to be one of the significant process parameter of sintering process for final densification as visible from Table 7. When we study the effect of sintering temperature on densification of SS316L, it is observed that the densification of SS316L powder begins at sintering temperature of about 1260 °C. The sintering begins slowly in the initial stage, but soon it causes rapid shrinkage in the intermediate stage; afterwards the shrinkage becomes much slower in the final stage to finish the sintering process [11]. At 1375 °C the SS316L powder begins to melt so the bondage among the particles becomes weak. But, contrary effects are observed in case of final density. The best results can be observed from Figure 6. The densification and changes in mechanical properties occur mainly in the heating period. Heating rate is also one of the significant process parameter of sintering process as observed from Tables 7. It affects both the densification and final mechanical properties of the sintered part. From Figure 7, it can be observed that best densification occurs at a heating rate of 12 °C/min. The sintering time is also a significant factor for densification and development of all

the mechanical properties and has the highest contribution as observed from Tables 7. During this time at sintering temperature the closed pores continue to reduce or vanish and the grain size increase. The holding time of 120 minutes is appropriate for obtaining the final components with high density, as visible from Figure 8. The cooling rate is also a significant factor for densification and mechanical properties but its contribution is low as compared to other parameters as observable from Tables 7. It can also be noted from Figures 8, that slow cooling rate is desirable and good results are observed at a cooling rate of 10 °C/min. The sintering atmosphere is an insignificant factor as observed from Tables 7.

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