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Research Paper

Effect of Temperature, Holding Time, and Addition of Sn on Density on Metal Injection Molding Sintering Process

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Abstract

Metal injection molding (MIM) is a metal forming technique that combines powder metallurgy with plastic injection molding. MIM is very efficient in manufacturing small and complex products in large quantities. The MIM process has four steps: mixing, debinding, injection molding, and sintering. This research was conducted to determine the effect of variations in Sn addition, temperature, and holding time on the density of Al-PP products after the sintering process. Density is mass per volume so to find out the volume of Al-PP products, the use of a 3D scanner was attempted along with the EinScan application and a mesh mixer. The Taguchi method was used for data processing to determine the influence of variations in Sn addition, temperature, and holding time on density. The calculation of the percentage contribution showed that variations in Sn addition, temperature, and holding time affected density by 47%, 21%, and 3%. Also, 2% Sn addition yielded a reasonably good microstructure formation compared to without Sn addition and 1% Sn addition, where many voids remained in the specimen (the more significant the voids, the lower the density).

Keywords: *3D scanner; metal injection molding; sintering; Sn; Taguchi.*

Introduction

Metal injection molding (MIM) is a net-shape or near-net-shape forming process with lower production costs and higher precision than other forming technologies. MIM is a metal-forming technique that combines powder metallurgy with plastic injection molding and is suitable for making large quantities of small specimens [1].

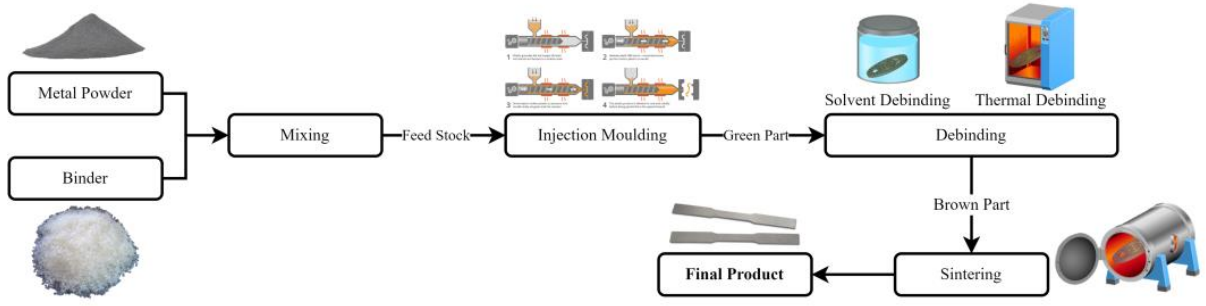

Figure 1 Metal injection molding (MIM) process

MIM can be done in mass production automatically with a relatively high level of precision. Figure 1 shows the MIM process, consisting of four processes: mixing, injection molding, debinding, and sintering. The first step consists of mixing the metal powder with a binder to produce feedstock as the injection raw material. The feedstock is heated and injected into the mold using an injection molding machine. The result of the injection

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process is called the green part. The green part is then heated in a chemical solution, which is called the solvent debinding process, or by heating the feedstock at a temperature above the decomposition of the binder component and holding it for a specified duration (holding time), which is called thermal debinding. The product of the solvent debinding or thermal debinding process is the brown part. The brown part is heated below the melting temperature of the metal to unite the particles to increase the product's strength and provide the necessary mechanical properties, which is called the sintering process [2].

In manufacturing the feedstock, aluminum powder was selected because it has good mechanical properties, corrosion resistance, and low density. Aluminum is usually applied in the automotive industry [3]. Low density can be overcome by adding tin (Sn), which benefits the sintering process by functioning as an activator for the growth of a microstructure that increases the strength of the metal [4]. Aluminum–tin (Al-Sn) is an alloy that has excellent corrosion resistance and surface finish [5].

The sintering process heats the brown part at a temperature below the melting point of the metal. The separated particles fuse so that the pores shrink and increase the strength of the metal powder [6]. The sintering process can cause an increase in density, strength, and shrinkage. As the sintering temperature increases, the shrinkage increases so that the porosity decreases and the hardness value increases. The density affects the formation of the resulting microstructure. The higher the density value, the fewer voids will be.

Liu *et al.* [7] concluded that magnesium blocks in the furnace are helpful for binding oxygen in the furnace. The study also found that adding 2% by weight of Sn and adding nitrogen in the atmospheric sintering process is beneficial for the micro-formation process, which greatly influences the resulting density, where density is the quotient between mass per volume.

Based on the research by Liu *et al.* [7], the present study was conducted to determine the effect of variations in Sn addition, temperature, and holding time on the density of the product in the sintered metal injection molding process. Density is one of the main factors determining the success rate of the MIM process; a higher density causes the resulting product to be more robust [8]. This study also aimed to find out the volume of the product based on volume measurements made with a 3D scanner.

Material and Method

Materials

The metal material used in this study was 45%-wt aluminum (Al) powder. The binder used to bind the aluminum powder during the injection molding process was 25%-wt PP (polypropylene) plastic with a melting point of 130 to 170 °C, 28 %-wt paraffin wax (PW), and 2%-wt stearic acid. The binder was mixed with the metal powder to make the feedstock. The solvent debinding process used a hexane solution with a boiling point of 65 to 70 °C and unreactive properties to the metal powder to help remove the binder. The advantage of this process is that the solution can be reused in the next procedure [9].

Method

The MIM process began with mixing the aluminum powder with the binder. The binder binds the metal powder and makes it more accessible during the injection molding process. Tin was used to make the feedstock. Then the injection molding process was carried out by inserting the feedstock into a hopper. After that, the material was melted and injected at pressure with a specific holding time to fill the mold and produce the green part. The dimensions of the product are shown in Figure 2. The solvent debinding process was applied to remove the binder by immersing the green part in the hexane liquid for a specific time duration and at a specific temperature. The brown part resulting from the solvent debinding process was sintered by heating it below the melting point of aluminum in order to form atomic bonds between particles to increase mechanical properties such as density, strength, and shrinkage.

The product's mass and volume were measured and the density before and after the sintering process was compared. The mass of the product was determined by weighing the product using a digital balance. A 3D scanner was used to find the volume. A 3D scanner is a device that can analyze a three-dimensional object and collect data that can be compiled into a three-dimensional model (point cloud geometry). Figure 3 is the

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application of the 3D scanning process, which used the EinScan application to display the projection results. After that, the volume of the product was found using the Meshmixer application.

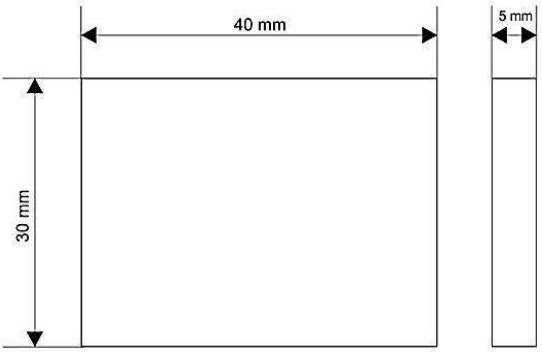

Figure 2 Dimensions of the product.

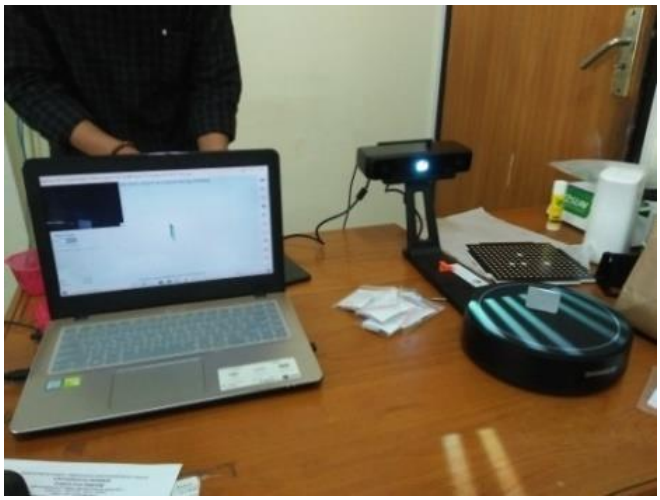

Figure 3 3D scanner.

In this study, analysis and evaluation of the parameters (factor variables) of the MIM process were carried out using the Taguchi method, which aims to optimize the product design and process so that the final result meets the targeted result with minimum variability. This is a quality control method to improve or maintain product quality as well as the quality of the process. The Taguchi experimental design uses an orthogonal matrix that aims to evaluate some process variables at a minimum number of experiments. Determination of the orthogonal matrix is based on the number of degrees of freedom by the number of process variables and the number of levels. The orthogonal matrix must have degrees of freedom greater than or equal to the degrees of freedom of the specified parameters. Therefore, this study used an orthogonal $L9(3)^3$ matrix with three replications using the Minitab 19 software [11]. The data was processed using ANOVA (analysis of variant) calculations, which is a data processing technique that analyzes statistically compiled experimental data. The effects of the variations in Sn addition, temperature, and holding time on the density were calculated using the Taguchi method. The Taguchi method uses the signal-to-noise (S/N) ratio to help identify independent variables that affect the experimental results [10].

Table 1 shows the variables used in the research to limit the research to be carried out based on the variables that significantly influence the process. The level selection for each variable was based on previous publications. In contrast, the factor selection was adjusted based on the tools used in the study using manual injection molding tools.

Table 1 Variables and levels used in the sintering process.

Code	Variables	Unit of measure	Level 1	Level 2	Level 3
A	Variation of Sn	-wt%	0	1	2

B	Temperature	°C	450	550	650
C	Holding Time	Hours	1	2	3

Result and Discussion

Data Optimization Results

Data from the experimental results were obtained based on the Taguchi experimental design with an L9(3)^3 orthogonal matrix using three replications. The independent variables, i.e., temperature, holding time, and Sn addition used in this study, were thought to affect the dependent variable (density). The following Eq. (1) was used to calculate the density reduction data resulting from the sintering process:

$$\text{Density Loss Percentage} = \left(\frac{Initial\ Density - Density\ After\ Sintering}{Initial\ Density}\right) x\ 100\% \quad (1)$$

The S/N ratio helps us know the independent variables that affected the experimental results. The characteristics of the S/N ratio consisted of smaller is better, nominal is best, and larger is better. The aspect of this study was that the less the density decreases, the better the quality, or, smaller is better. Eq. (2) illustrates the calculation of the effect of the S/N ration on the density percentage, where Y_i denotes the density decrease in experiments 1, 2, and 3. Meanwhile, *"n* is the number of experiments carried out, with the following Eq. (2):

$$\text{S/N} = -10\ log\left[\sum_{i=1}^{r}\frac{Y_i^2}{n}\right] \quad (2)$$

The density reduction percentage and the S/N ratio calculations are shown in Table 2 for each combination of three repetitions.

Table 2 Experimental data on density loss percentage.

Control Factor			Density Decrease (%)			Density Loss Percentage (%)	S/N Ratio
Variation of Sn (%)	Temperature °C	Holding Time (hr)	Experiment 1 (%)	Experiment 2 (%)	Experiment 3 (%)		
0	450	1	43	40	40	41	-32.2557
0	550	2	35	31	33	33	-30.3703
0	650	3	30	30	28	29	-29.2480
1	450	2	43	33	31	36	-31.1261
1	550	3	30	31	30	30	-29.5424
1	650	1	28	28	25	27	-28.6273
2	450	3	29	28	28	28	-28.9432
2	550	1	25	27	25	26	-28.2995
2	650	2	25	24	23	24	-27.6042

The requirement for analysis of variance is that the residual must meet the IIDN assumption (0, σ2). It must be normally distributed with a mean of zero and a specific variance value. Testing was carried out using the Minitab 19 application. Normal distribution testing, or normality testing (0, σ2), was conducted using the Kolmogorov-Smirnov normality test. The hypotheses used in this test were:

H0: Residues are normally distributed.

H1: Residues are generally not distributed.

H0 is accepted if P > α = 0.05

Figure 4 shows the results of the data tested using the Kolmogorov-Smirnov normality test, namely P > α = 0.150, which means that the value is more significant than α = 0.05. This means that H0 is, which means that normally distributed residuals have been reached.

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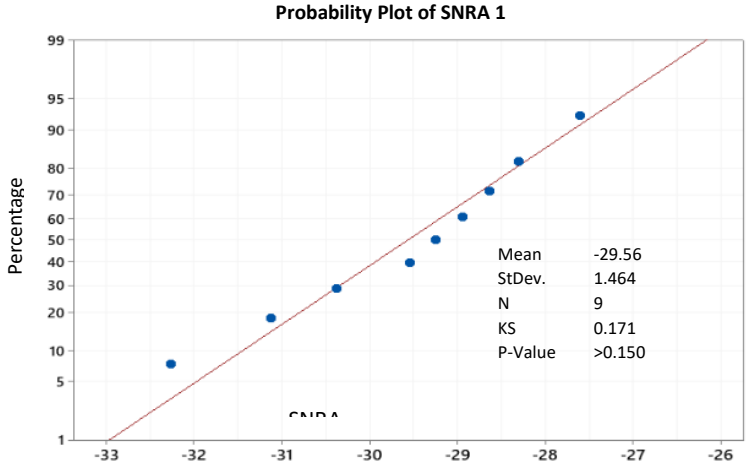

Figure 4 Normal distribution test plot

The Taguchi method uses the calculation of the S/N ratio. Using the procedure for dividing the total S/N ratio at each level by the number of levels of each parameter, as stated in Eq. (3), Table 3 gives the average S/N value for each level:

$$\bar{x} = \frac{\sum_{i=0}^{n} S/N\ Ratio}{n} \tag{3}$$

Table 3 Average response value of S/N ratio on each level parameter.

Parameter	Level 1	Level 2	Level 3	Difference (max-min)
Variation of Sn	-30.6246	-29.7653	**-28.2823**	2.3424
Temperature	-30.7750	-29.4041	**-28.4932**	2.2818
Holding Time	-29.7275	-29.7002	**-29.2445**	0.4830
Average		-29.5574		

A plot of the S/N ratio value at each level of the parameters Sn addition, temperature, and holding time can be seen in Figure 5. The optimum parameter level shows the highest S/N ratio value. From the calculation results in Table 3, it can be concluded that the parameters Sn addition and temperature in the sintering process significantly affected the density, whereas the holding time had no significant effect.

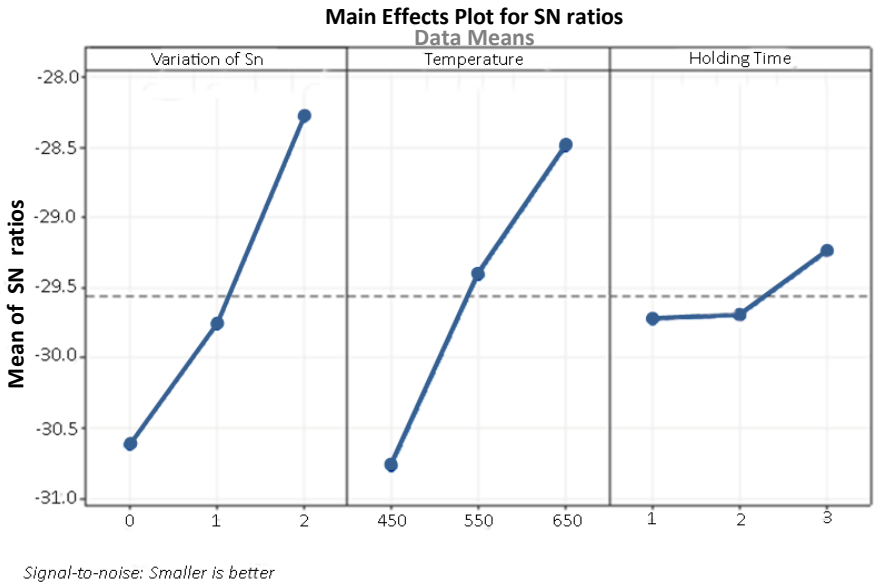

Figure 5 Plot for S/N ratio to density.

Analysis of Variance Test (ANOVA)

The aromatic production from methanol feedstock has almost reached industrial maturity, especially in China. (ANOVA) was used to help determine the parameters that significantly affect the response under study [12]. The following is the calculation procedure of ANOVA with the value of the S/N ratio:

1. Calculating the total number of squares:

$$SST=\sum_{i=1}^{n}(yi-\bar{y})^2 \quad (4)$$

where *yi* is the value of the S/N ratio and $\bar{y}$ is the average value of the S/N ratio.

2. The sum of the squares of each parameter:

$$SSA=\sum_{i=1}^{n}(Ai-\bar{y})^2 \quad (5)$$

where *nA* is the number of levels in each parameter, *Ai* is the average value of the S/N ratio at each level, and $\bar{y}$ is the average value of the S/N ratio at all levels.

3. The mean square of the middle:

$$MSA=\frac{SS_A}{do_A} \quad (6)$$

SS_A is the sum of the squares of each parameter, and Df_A is the degree of freedom.

4. Contribution percentage calculation:

$$SS'_A=SSA-df_A\,.\,MS_{Res} \quad (7)$$

$$P_A=\frac{SS'A}{SST} \quad (8)$$

SS_A is the sum of the squares of each parameter, df_A is the degree of freedom, MS_{Res} is the residual error value, and *SST* is the total number of squares.

The calculation of the percentage contribution shows how much influence the parameters had on the dependent variable under study. If the percentage value of the residual contribution was less than fifteen percent, then all parameters affected the dependent variable. In contrast, if the residual contribution percentage was more than fifteen percent, then some parameters had a negligible effect on the dependent variable. The following are the outcomes of the calculation of the ANOVA value's contribution to the S/N ratio value displayed in Table 4.

Table 4 ANOVA results and parameter contributions by calculating the S/N ratio on density.

Parameter	DF	SS	MS	F-value	Percentage
Sn addition	2	8.4244	4.2122	23.6604	47%
Temperature	2	7.9158	3.9579	22.2321	21%
Holding time	2	0.4416	0.2208	1.2403	3%
Residual error	2	0.3561	0.1780		29%
Total	8	17.1378			

From the calculation results, the variables that influenced the dependent variable had an F_{count} (F-value) greater than F_{table}. In this study, the significance level used was 5 %, where the value of F_{table} was ($F_{0.05;2;2}$) = 19.00 [11]. If the initial hypothesis (H_0) and alternative hypothesis (H_1) are used as a hypothesis test using an F distribution, then:

1. If $F_{count} > F_{table}$, H_1 is accepted and H_0 is rejected, which means that the parameters significantly affect the density.
2. If $F_{count} < F_{table}$, then H_1 is rejected, and H_0 is accepted, which means the parameters do not significantly affect the density.

Thus it can be concluded that, based on the hypothesis test with an F distribution, two parameters had a significant effect on the resulting density value, and one parameter did not. As shown in Table 5, below:

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Table 5 Hypothesis condition H_0.

Symbol	Control Factor	H_0 condition
A	Variation of Sn	Rejected
B	Temperature	Rejected
C	Holding Time	Accepted

Density

Variation Parameter Sn

The findings of the hypothesis test using an F distribution showed that addition of Sn in the sintering process significantly affected the density and addition of more Sn caused the density value to increase more. Liu *et al.* [7] stated that addition of Sn yielded a higher density than no addition of Sn, as shown in Figure 6.

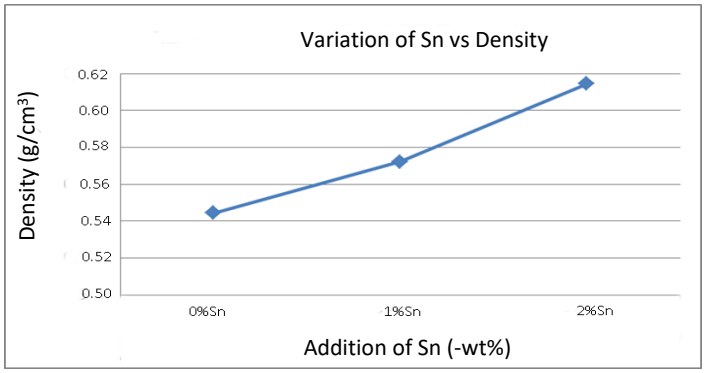
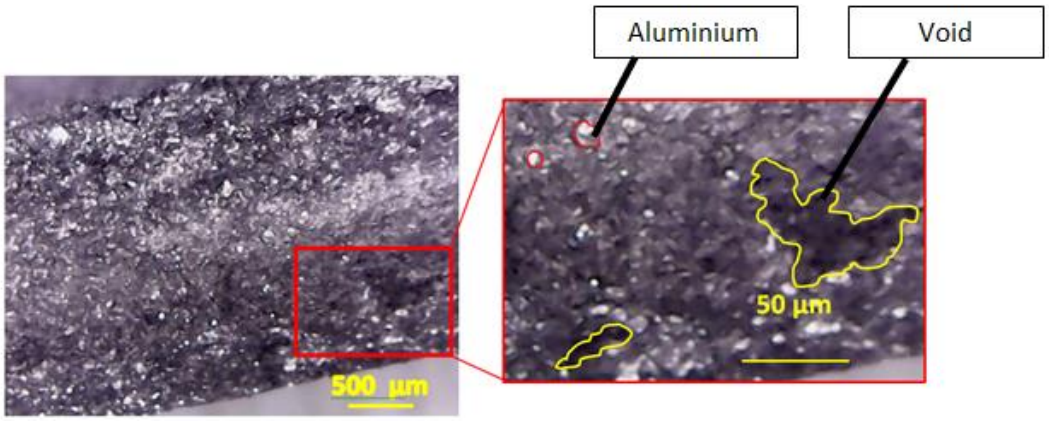


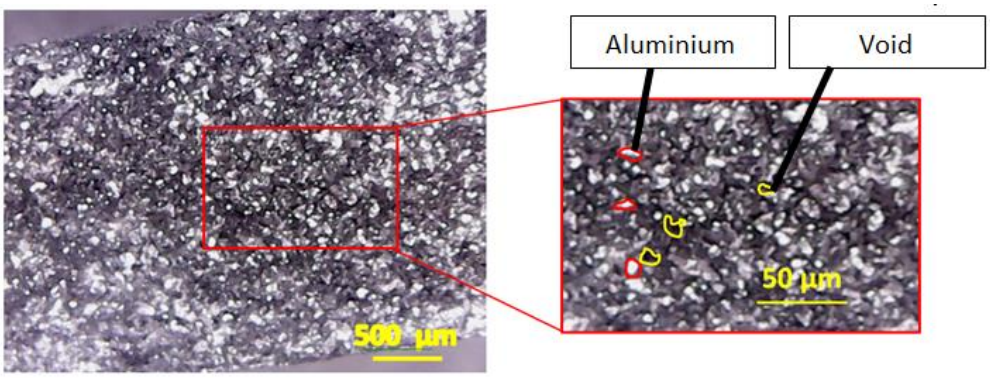
Figure 6 Diagram of density vs variation of Sn.

This can be seen based on statistical calculations with a percentage contribution value of 47% in Table 4, with the most optimal level being level 3 (2% Sn addition). When viewed visually, the products without Sn and 1% Sn addition do not have a perfect microstructure, with still many voids in the specimen. The more voids, the lower the density, as shown in Figure 7. Figure 7(c) shows that there was a separation between the aluminum metal, the voids and the remaining binder (PP), which will most likely result in variations in density in the product. This separation can occur because there is non-uniform shrinking in each product zone [13]. Solutions to reduce this problem need to be further investigated.

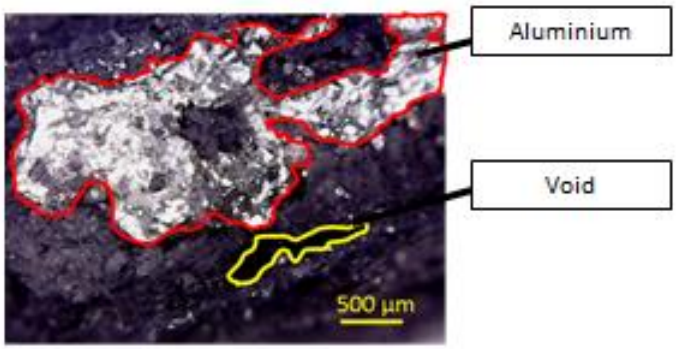


(a)

Figure 7 (a) Surface appearance of the 0% Sn specimen. It can be seen that there is a fairly large distribution of aluminum and voids. (b) Surface appearance of the 1% Sn specimen. The voids are still spread out but not as widely as without Sn addition. (c) The addition of 2% Sn shows a conglomeration of aluminum and a separation of voids.



(b)



(c)

Figure 7 Continued. (a) Surface appearance of the 0% Sn specimen. It can be seen that there is a fairly large distribution of aluminum and voids. (b) Surface appearance of the 1% Sn specimen. The voids are still spread out but not as widely as without Sn addition. (c) The addition of 2% Sn shows a conglomeration of aluminum and a separation of voids.

Temperature Parameter

The temperature is a controlling factor that significantly affects the increase in density. This can be seen from statistical calculations, with a percentage contribution value of 21 % and the optimum level occurring at level 3 and a temperature of 650 °C. This result supports the assertion made by Kent *et al.* [14] that the density increases with increasing sintering temperature, as illustrated in Figure 8.

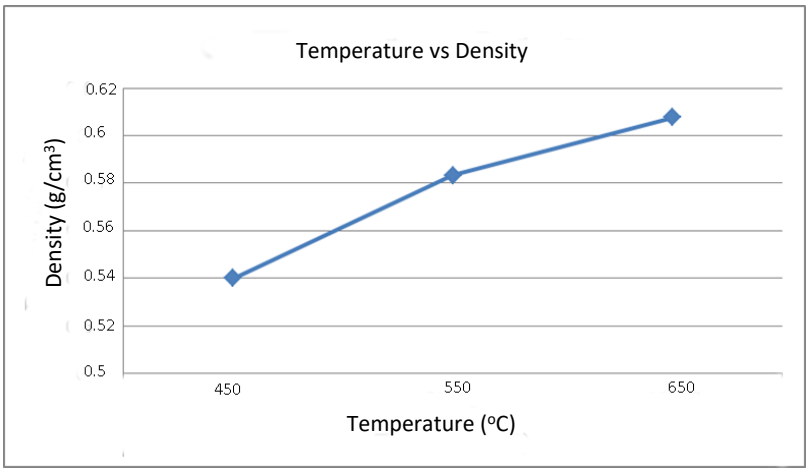


Figure 8 Diagram of density vs temperature.

Holding Time

Holding time is a parameter that had no significant effect on the density of the sintering process, because the percentage contribution value was only 3% (Table 4)**.** The distribution was F_{count} = 1.2403 < (F0.05;2;2) = 19.00. Therefore, H1 is rejected and H0 is accepted, which means that the holding time parameter has an insignificant effect on density. If the holding time is longer, the microstructure formation is still intact and causes low density. In this study, the optimal variation of holding time occurred at level 3 (3 hours).

Conclusion

Based on the analysis of variance (ANOVA), it can be concluded that the parameters of Sn addition, temperature, and holding time affect the Al-PP product. The materials used in this research were a mixture of aluminum, polypropylene, paraffin wax, and stearic acid. The calculations indicate that differences in Sn of 47%, in temperature of 21%, and in holding time of 3% all impacted the percentage contribution to density. The optimal value for Sn addition was level 3 (2%). The optimal temperature was level 3 (650 °C). Meanwhile, the holding time had no significant effect on the density, because the contribution value was only 3%. The optimal holding time was level 3 (3 hours).

References

[1] Ji, C.H., Loh, N.H., Khor, K.A. & Tor, S.B., *Sintering Study of 316L Stainless Steel Metal Injection Molding Parts Using Taguchi Method: Final Density*. Material Science and Engineering, **A311**(1-2), pp. 74-82, 2001. doi:10.1016/S0921-5093(01)00942-X.

[2] Brown, J., *Modern Manufacturing Processes*, First edition. New York: Industrial Press Inc., 1991.

[3] Asmawi, R., Ibrahim, M.H.I., Amin, A.M., Mustafa, N. & Alawi., N., *Mixing Study of Alumunium Waste as Metal Powder for Waste Polystyrene Binder System in Metal Injection Molding (MIM)*, Applied Mechanics and Materials, **660**, pp. 239-243, 2014. doi:10.4028/www.scientific.net/AMM.660.239.

[4] Ni, J., Yu, M. & Han, K., *Debinding and Sintering of an Injection-Moulded Hypereutectic Al-Si Alloy*. Materials, **11**(807), pp. 1-10, 2018. doi:10.3390/ma11050807.

[5] Sowiyk, H.P. & Bayuseno, A.P., *The Effect of the Addition of Tin (Sn) Elements on Physical and Mechanical Properties in Bearing Materials Based on Aluminum (Al) Results of HPDC Casting*. Journal of Mechanical Engineering S-1, **4**(3), pp. 290- 298, 2016.

[6] Schlieper, G., Dowson, G., Williams, B. & Petzoldt, F., *Metal Injection Moulding, A Manufacturing Process for Precision Engineering Components*. Shrewsbury: European Powder Metallurgy Association (EPMA), 2013.

[7] Liu, Z.Y., Sercombe, T.B. & Schaffer, G.B., *Metal Injection Moulding Aluminium Alloy 6061 with Tin*. Powder Metallurgy, **51**(1), pp. 78-83, 2008. doi: 10.1179/174329008X284859.

[8] Wahi, A., Muhammad, N., Sulong, A.B. & Ahmad, R.N., *Effect of Sintering Temperature on Density*, Hardness and Strength of MIM Co30Cr6Mo Biomedical Alloy. J. Jpn. Soc. Powder Metallurgy, **63**(7), pp. 434-437, 2016. doi: 10.2497/jjspm.63.434.

[9] Jaman, W.S., Virdhian, S. & Afrilinda, E., *Preliminary Study Solvent Debinding Feedstock Metal Injection Molding Fe2%Ni*, Metal Indonesia, **40**(1), pp. 10-16, 2018. (Text in Indonesian) doi:10.32423/jmi.2018.v40.10-16.

[10] Ghani, J.A., Choudhury, I.A. & Hassan, H.H., *Application of Taguchi Method in the Optimization of End Milling Parameters*. Journal of Material Processing Technology, **145**(1), pp. 84-92, 2004. doi:10.1016/S0924-0136(03)00865-3.

[11] Soejanto, I., *Experimental Design Using the Taguchi Method*, Yogyakarta: Graha Ilmu, 2009.

[12] Kamaruddin, S., Khan, A. & Foong, S.H., *Application of Taguchi Method in the Optimization of Injection Moulding Parameters for Mnaufacturing Products from Plastic Blend*. IACSIT International Journal of Engineering and Technology. **2**(6), pp. 574-580, 2010.

[13] Butković, S., Šarić, E. & Mehmedović, M., *Porosity Distribution in Metal Injection Molded Parts.* Advanced Technologies & Materials*,* **46**(1), pp. 7-10, 2021. doi:10.24867/ATM-2021-1-002.

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[14] Kent, D., Liu, Z.Y. & Schaffer, G.B., *Powder Injection Moulding of an Al-AIN Metal Matrix Composite*. Material Science and Engineering, pp.352-356, 2009. doi:10.1016/j.msea.2009.02.001.

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