Optimizing Sintering Process to Produce Highest Density of Porous Ti-6Al-4V

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ABSTRACT

Metal Injection Moulding (MIM) is a promising approach to produce to a near net-shape product of intricate geometry with cost-effective production. Nowadays, several researches have been done on implementing MIM to manufacture products by applying Titanium and its alloy as a main subject. Titanium and its alloys are renowned materials with good mechanical properties and high corrosion resistance. This study presents an optimization of sintering parameters for the highest density of porous Ti-6Al-4V powder mixed with 60wt% of palm stearin and 40wt% of polyethylene by metal injection moulding (MIM) technique. The density of the sintered part was resulted from tremendous densification of the sample. Sintering parameters have been optimized using Taguchi method of L9 (34) orthogonal array. The result from Taguchi method, combination of A2, B2, C1 and D1 as the best set of factors. This means that the sintering temperature at 1300oC; sintering duration, 120 min; heating rate 4oC/min and cooling rate 9oC/min were the optimum level that could statistically result in produce highest density of porous. The analysis of variance (ANOVA) was employed to determine the significant level (α) and its contribution to the variables of the final density. The study demonstrated that sintering temperature was the most influential variable contributed to the best final density, followed by sintering duration, cooling rate and heating rate.

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Introduction

Powder Metallurgy (PM) is a proven of advanced and sophisticated technology for producing net shape metal components in every imaginable design [1-3]. It can manufacture a product in any configuration of sizes, ranging from tiny to huge. PM offers component designers and product manufactures a lot of advantages when compared to other metal forming processes. It makes possible complex or unique shapes that would otherwise be impractical r even impossible. It also reduces machining as well as scrap. There a four main processing procedures in MIM which are materials mixing, injection moulding, debinding and sintering [4-5]. MIM starts by mixing selected metal powder (usually Titanium and its alloys) at certain size (μm) and binders at appropriate 2 compositions to synthesize a mixture called a feedstock. This step is a vital process in MIM. The binders provide lubrication during the injection by uniformly coating the metal powder and filling all the gaps between the powder particles [6-7]. Binder usually used is polyethylene (PE) or recently alternative for multi-components conventional binder system, Palm Stearin (PS) [8-10].

Next, the mixture is now feedstock is poured into a hopper and the injection molding process will take place. The machine is controlled by certain parameters (pressure, heating temperature, heating rate, speed of the injecting screw, cooling rate). Then the feedstock is injected into a specific-designed mold cavity [11-13]. Then, the product is now called green part (plastic metal). The green part will undergo debinding process, a process of which the plastic part will be removed from the metal [14]. After debinding process is finished, the part is now called brown part because of the fragile pore structure (gaps between metal particles). The brown part requires the metal to be condensed in a furnace process called sintering [15-17]. After sintering, grey part is produced that has comparable mechanical and physical properties with parts made using classic metalworking methods.

Methodology

Material Preparation: Feedstock

Metal Powder Ti-6Al-4V

Sources from the supplier TLS Technik GmbH used in this study shows the characteristics of the metal powder Ti-6Al-4V as shown in Table 1 while Table 2 shows the chemical content of the metal powder. Powder metal Ti-6Al-4V used in this study was sphere-shaped generated through the gas atomization. Figure 1 shows a Scanning Electron micrograph (SEM) of metal powder Ti-6Al-4V.

Table 1: Characteristics of Ti-6Al-4V

Particle shape	Sphere
Size	<35 µm
Picnometer Density	4382.7 kg/m3
Melting	1650°C

Table 2: Chemical Composition of Ti-6Al-4V

Al	V	C	Fe	O	N	H	Ti(wt%)
5.99	4.08	0.005	0.043	0.185	0.004	0.002	Bal

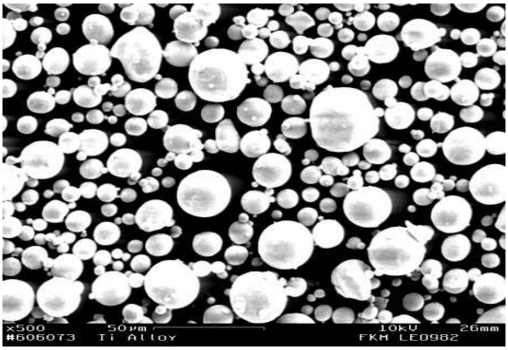

Figure 1: SEM particle of Ti-6Al-4V

Binder

The binder used in this study consists of PS which acts as a main binder (primer) while PE is a backbone binder or secondary binder. The density of PS used in this study was 0.891g/cm3, while the density of PE is 0.91g/cm^3. The composition of the binder used in this study is based on 60% PS and 40% PE were determined based on the percentage of the mass. This composition was chosen because it is stable based on a study conducted by Omar et. al. [3] which applies the binder with metal powder 316L stainless spherical.

Apparatus and Method of Experiment

Mixing Process

The mixing process is a process for the preparation of a homogeneous feedstock. Feedstock prepared by mixing metal powder Ti-6Al-4V with a binder. Z-blade mixer Winkworth was used to produce feedstock. Feedstock components were firstly weighed by a certain percentage and the mixing process is carried out at temperatures 150°C which is above the melting temperature of PE. PE was then poured into the mixing chamber until the PE melted followed by metal powder Ti-6Al-4V and later binder PS was poured into the mixing chamber. The mixing process is carried out for one hour.

Granulation Process

This process involves the crushing machine to obtain the feedstock in the form of small grains as shown in Figure 3.4. Feedstock produced from the mixing machine is then inserted into the crushing machine to be crushed from the dough into small granules.

Injection Molding Process

The injection was performed using the injection molding machine Battenfeld BA 250 CDC as shown in Figure 3.5. The injected body is in tensile bar-shaped, as shown in Figure 3.6. Injection process was started after the preset parameters are achieved. Each combination of parameters is carried out by five sample injection.

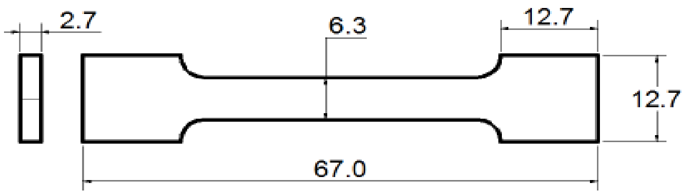

Figure 2: Tensile bar sample

Debinding Process

The dried brown samples were next put into a vacuum furnace to undergo the thermal pyrolysis process begins at a temperature of 505oC for 90 minutes and followed by a sintering process.

Table 3: Sintering Parameters

	Parameter	Level		
A	Sintering Temperature (°C)	1200	1250	1300
B	Sintering Time (min)	60	90	120
C	Heating Rate (°C/min)	3	4	5
D	Cooling Rate (°C/min)	8	9	10

Sintering Process

This process is the most important process which needs to be highly focused on because the parameters for the scope of the study will impact the changes to as-sintered part generated after sintering process. The parameters that were controlled in this process were the sintering temperature, sintering time, heating rate and cooling rate as shown in Table 3. The Sintering process was carried out in a high vacuum furnace type VAC-TEC model VTC 500HTSF shown in Figure 3. Vacuum pressure that can be achieved by this furnace is up to 9.5x10-6 mbar or 10-5 torr.

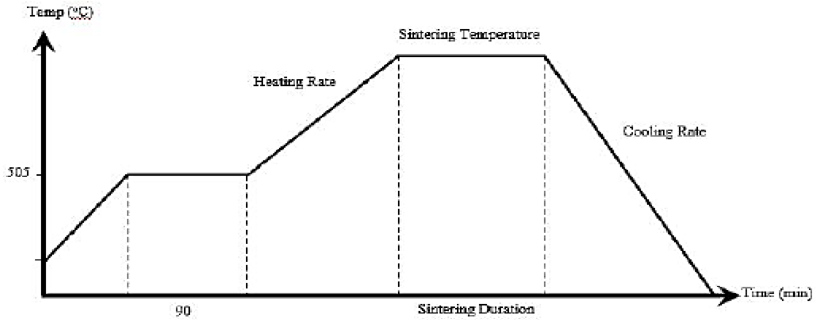

Figure 3: Pyrolysis graph and heating cycle sintering process

Design of Experiment (DOE)

In Taguchi Method of Optimization, the total degree of freedom needs to be computed in order to select an appropriate OA for the experiments. Basically, the degree of freedom (DOF) for the OA is greater or at least equal to those of process parameters [18-19]. In this paper, four sintering process parameters were investigated i.e. sintering temperature, sintering duration, heating rate and cooling rate. The selection sintering parameters along with their levels are given in Table 4. The total DOF for four factors, each at three levels is 8. Therefore, OA of L9 is the most suitable for the DOE which consist of 9 experiment trials and 4 columns of parameters. Each experiment was conducted with three replications.

Table 4: Injection Parameters for Three Level Taguchi Design

Level	Sintering Temperature (°C)	Sintering Duration (min)	Heating Rate (°C/min)	Cooling Rate (°C/min)
	A	B	C	D
0	1200	60	3	8
1	1250	90	4	9
2	1300	120	5	10

Result and Discussion

Removal of Binders and Space Holder from Green Parts

Removal of binders PE and PS and space holder, NaCl were conducted separately. Firstly, the process of solvent extraction was taken place where PS was removed by soaking the green part in heptane. Secondly, NaCl was removed by immersing the green part in distilled water. The process is known as water leaching. Thirdly, thermal debinding took place to remove the PE. In that process, the specimen was loaded in a vacuum furnace heated above the decomposed temperature of PE. Usually, thermal debinding is combined with sintering process to uphold the debound structure from rupture due to weak bonding of metal powder particles [20]. Figure 4 shows comparison before and after solvent debinding and water leaching process. PS and NaCl shown before removal; are clearly removed after removal of the binder and space holder through solvent debinding process

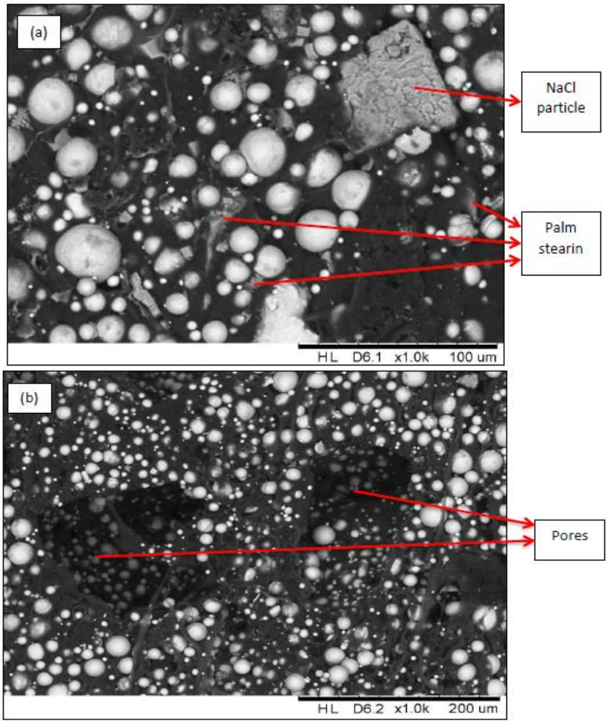

Figure 4: Solvent debinding and water leaching process, a) before and b) after.

Thermal Debinding and Sintering

Thermal debinding and sintering was conducted in a high vacuum furnace to prevent oxidation or other impurities picked up by the samples. In both process, several parameters were assigned to ascertain the success of thermal debinding and sintering process. Those parameters are temperature, sintering time, heating rate and cooling rate. However, the most crucial parameter need to be controlled in this process was the heating rate of thermal debinding since PE needs to be removed gently from the green specimen and at the same time maintaining the structural integrity of the specimen.

In order to ensure PE is removed completely, the temperature of thermal debinding was set above the decomposed temperature of PE based on thermal analysis of the binder. Meanwhile, sintering temperature was set lower than Ti-6Al-4V particles melting point to allow enough sufficient grain growth and to prevent over shrinkage of as sintered products [1]. After the success of thermal debinding and sintering process of tensile bar specimen, the dimension of the as-sintered part was measure to determine the shrinkage. Theoretically, during sintering process, the particles of metal powder will bind together to form a strong bonding. As the metal powder particles binding together, the whole structure experienced shrinkage because the reinforcement particles have 'fill in the blank' behaviour after the removal of binder and space holder. Figure 5 shows the shrinkage of the as-sintered sample compared to green part and brown part

Figure 5: Shrinkage effect before and after sintering (a) green part (b) PS and NaCl debound part (c) as-sintered part

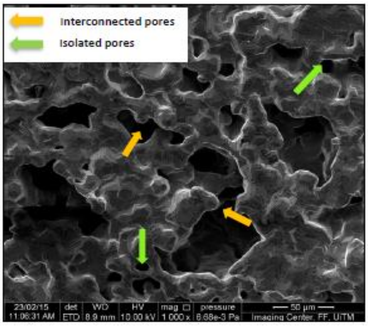

Figure 6: The example of interconnected pores and isolated pores in the as-sintered specimen.

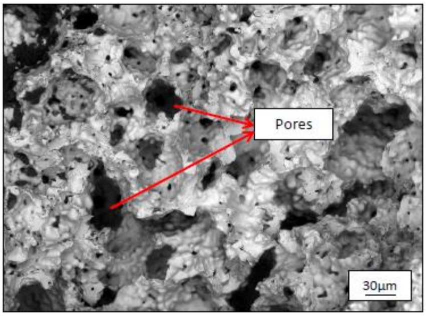

Figure 7: The porosity that affects the mechanical strength of the sintered specimen.

Signal-to-Noise (S/N) Ratio

Archimedes method used to calculate the density based on the principle of water float. In this situation, there were no emissions of air bubbles during the grey part submerged in the water compared during density measurement of the grey part. This was the result due to the pores that exist already been closed as a result of shrinkage of the powder and grain production. Therefore, a higher density were produced and approaching the theoretical density. The S/N ratios were computed for each of 9 trial conditions. The density is a "the higher the better" type of quality characteristic. So the S/N ratio for that type of response was used as given by:

$$S/N=-10\log\left[\frac{1}{n}\sum_{j=1}^{n}\frac{1}{Y_{ij}^{2}}\right]$$

where Y_{ij} is the amount of score for the density of porous and N is the total number of shots for each trial.

Table 5: Taguchi's L9 (34) orthogonal array (OA) demonstrates the density of grey part and the experimental trials

	Parameter				Density of Grey Part			
Trial	A	B	C	D	1	2	3	S/N
1	0	0	0	0	3.3215	3.3216	3.3303	10.43442
2	0	1	1	1	3.3956	3.3888	3.3854	10.6038
3	0	2	2	2	3.3712	3.4134	3.3112	10.53836
4	1	0	1	2	3.2672	3.2743	3.2901	10.30995
5	1	1	2	0	3.3261	3.2678	3.2577	10.32662
6	1	2	0	1	3.2991	3.3587	3.3561	10.46875
7	2	0	2	1	3.3487	3.4011	3.4065	10.59151
8	2	1	0	2	3.3985	3.3912	3.3467	10.57465
9	2	2	1	0	3.4124	3.4099	3.3984	10.64715
							Σ	94.4952
							T	10.49947

The experimental results for the density of porous characteristic and corresponding *S/N* ratio using above is shown in Table 5 and the *S/N* response was plotted in Figure 8. The optimum set of factors can be formulated by selecting the level with the highest S/N value. The result is a combination of A2, B2, C1 and D1 as the best set of factors. This means that the sintering temperature, 1300oC; sintering duration, 120 minutes; heating rate, 4oC/min; cooling rate, 9oC/min are the optimum level. The highest densities obtained were 3.4134 g/cm3 and lowest density obtained were 3.2577 g/cm^3.

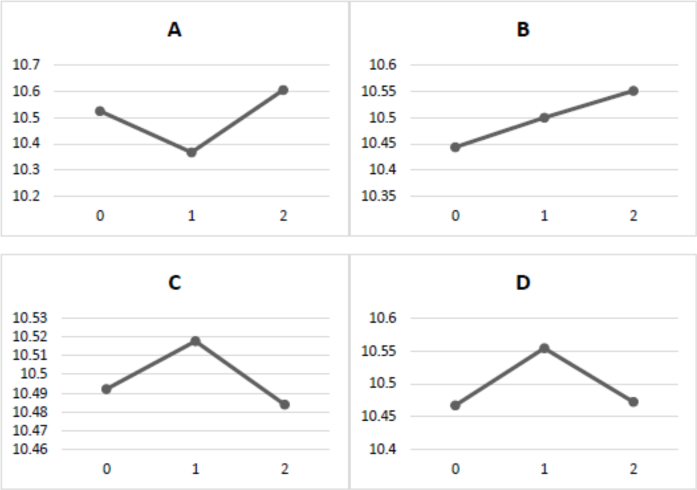

Figure 8: Signal-to-Noise: Larger is better

Analysis of Variance (ANOVA)

The aim of ANOVA is to evaluate the significant of the process parameters to the as-sintered part density. The results of ANOVA analysis of raw data for higher green density are shown in Table 6.

From Table 6, it can be seen that F18, 2, 0.01 is 2.62 for a level of significance equal, α = 0.1 (or 90% confidence level). It is shown that the F-value of factor A which is 3.266439 is greater than F18, 2, 0.01 = 2.62. Factor A indicates the most critical parameter for the best density of porosity characteristic, followed by factor B, factor D and factor C. Thus, during sintering process, sintering temperature needs to give more attention. Factor C are not significant affecting for high density of porosity even though it give contribution during sintering process but the confidence level are less than 90%.

Table 6: ANOVA of as-sintered part

Variable	Factor	DF	Sum Squared	Variance	F	% Contribution
A	Sintering Temperature	2	0.0288655	0.014433	3.26644	24.2
B	Sintering Duration	2	0.0056388	0.002819	0.63809	4.73
C	Heating Rate	2	0.0006762	0.000338	0.07652	0.57
D	Cooling Rate	2	0.0045859	0.002293	0.51895	3.84
Error		18	0.0795329	0.004419		66.66
Total		26	0.1192993			100

Table 7: Significance level for each factor with DOF = 2 and error = 18 based on F Distribution Table

Significance level	F-Value	Factors and It's F-Value
$\alpha = 0.1$ (90% confidence level)	2.62	A (3.266439)
$\alpha < 0.1$ (< 90% confidence level)	< 2.62	B (0.638092) C (0.076524) D (0.518945)

Conclusion

Optimization parameters in injection moulding process of Ti-4V-6Al powder using binders of PS and PE to produce highest density of porosity were successfully investigated through Taguchi Method. The optimum sintering parameter were found to be sintering temperature at 1300oC; sintering time, 120 minutes; heating rate, 4oC/min; cooling rate, 9oC/min are the optimum level. Based on ANOVA results, sintering temperature (Factor A) is the most significant effect which shows a contribution of 24.2%, followed by the sintering duration (factor B) at 4.73%, cooling rate (factor D) at 3.84% and heating rate (factor C) at 0.57%. Factor A is highest contributor into producing highest density of porosity Ti-6Al-4V because it's F-value is 3.266439 which is above than F-value of level of significance equal, $\alpha = 0.1$ or 90% confidence level which is 2.62

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