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Sintering Parameter Optimization of Ti-6Al-4V Metal Injection Molding for Highest Strength Using Palm Stearin Binder

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Abstract

This paper presents an optimization of sintering parameters for the best sintered strength of Ti-6Al-4V powder mixed with 60wt% of palm stearin binder and 40wt% of polyethylene by metal injection moulding (MIM) technique. The mechanical properties of the sintered part are resulted from tremendous densification of the sample. Sintering parameters have been optimized using Taguchi method of L_9 (3^4) orthogonal array. The analysis of variance (ANOVA) was employed to determine the significant levels (α) and its contribution to the variables of the final strength. The study demonstrated that sintering temperature was the most influential variable contributes to the best final strength, followed by heating rate, dwelling time and cooling temperature. Based on these results, samples displayed yield strength of 934.3MPa, and a plastic elongation of more than 10% were produced. These values meet the requirements of the ASTM B348-02 for titanium alloy medical grade.

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Keywords: Ti-6Al-4Vpowder, Palm stearin, Metal injection molding, Taguchi method

1.Introduction

Titanium and its alloys have become very trendy materials because of their low density, high corrosion resistance and excellent mechanical properties [1-2]. Nowadays, the efficacy of titanium for medical implants, for exclusive sporting gears and also for jewelry is recognized. Titanium parts are still expensive not only because of high raw materials price but also because of complexity forming, machining and welding [3]. One possible process candidate that can potentially provide a reduction of the manufacturing costs is the metal injection molding (MIM) technique. MIM processing offers several unique advantages for the mass production of near net shape components [4-5]. The MIM process comprises of four main steps: mixing, injection molding, debinding and sintering [6].

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Generally for Ti-MIM, the selection of proper binder is a prerequisite for producing complete parts successfully. Several attempts of binder formulations have been made in Ti-MIM which normally are wax-based binder systems and the common backbone polymers are polyethylene and polyporpylene. Various waxes and acids are also added to the binder to lower down the viscosity and increase the wettability and miscibility [7].On the other hand, thisresearch describes the Ti-MIM process which employs a palm stearin binder as a major component with reduced decomposing substances. Palm stearin is best known for its ability to provide a capillary route for the removal of remaining binder in the later stage of debinding prior to sintering [8]. The binder system, was first reported by Iriany et al. (2002) after they developed a binder made of a major fraction of palm stearin and a minor fraction of polyethylene used 316L stainless steel powder as a test material. Other uses of palm stearin as the primary solvent extraction component have been reported by Istikamah et al. (2007) where the palm stearin aided in designing feedstock systems with powder 316L stainless steel aimed at maximizing the ease of processing and shape retention during debinding and sintering, while minimizing processing times.

The problem of contamination with oxygen, nitrogen and carbon during debinding and sintering operations, which strongly influences the mechanical properties should be largely be overcome. Therefore in order to reduce contamination of Ti with carbon and oxygen due to debinding, the requirement is to reduce the amount of decomposable substances in binder.Alternatively, part based on Ti-6Al-4V with reasonable strength-levels and large plastic elongations must be achieved. This study aimed to determine the feasibility of palm stearin as a binder which not generous any contamination of the sintered part.

In this study, the process parameters for the strength of the sintered part were optimized using Taguchi method. Taguchi methods have been used widely in engineering analysis to optimize the performance characteristics through the setting of design parameters. Taguchi method is also strong tool for the design of high quality systems.A model based on L_9 orthogonal array of Taguchi design was created by utilizing the S/N ratio and ANOVA for the optimization process. The effects of four sintering factors: sintering temperature, dwell time, heating rate, and cooling time on the final density were investigated. The optimum sintering condition was proposed and confirmation experiments were conducted.

1. Materials and Method

Ti-6Al-4V used in this study was supplied by TLS TechnikGmBH, Germany with average powder particle size 18μm.

Fig. 1 shows the morphology of the Ti-6Al-4V alloy powder. The particle of Ti-6Al-4V alloy in spherical shape which it pycnometer density of 4.38 g/cm^3 was mixed with 60wt% of palm stearin and 40wt% of polyethylene. The powder-binder were mixed in Z-blade mixer at temperature 150 ^{o}C during one hour to produce feedstock of 65vol.%. The feedstock was injected using Battenfeld, BA 250 CDC injection molding machine. Fig.2 shows the schematic diagramof the Ti-MIM tensile bar with thickness of 2.7 mm.The debinding stage was divided into solvent and thermal debinding. Heptane was used to remove palm stearin for 6h. The remaining binder, polypropylene was removed using a debinding Split Furnace modelRS800/200/200at 550 ^{o}C for 4h.

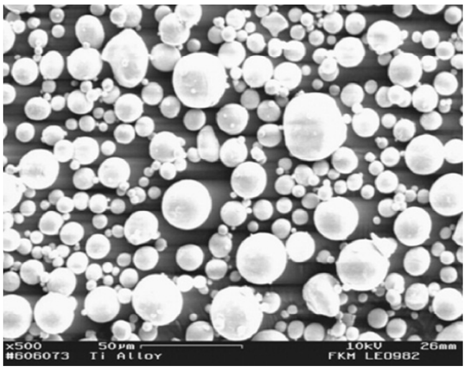

Fig. 1. Scanning electron micrograph showing the morphology of the Ti-6Al-4V.

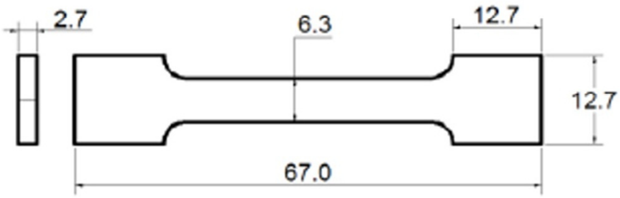

Fig. 2. Injection mold part and geometry. Dimensions are in mm.

Optimization for the injection parameter has been studied by Mohamad Nor et al. resulting a significant optimum injection parameter for MIM feedstock. As a consequence to the injection parameter optimized by those literatures, this paper presents a sintering parameter optimization which utilizes the optimized injection parameter. The green molded specimens were subjected to a solvent extraction step where two third of the binder system was removedby immersed into the heptanes for 6 hours at the temperature of 60°C.

In this paper, L_9 (3^4) orthogonal array consisting of 9 experiment trials and 4 column was 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. Strength of the sintered parts is to be optimized in this study. The high vacuum furnace Korea VAC-TEC, VTC 500HTSF with vacuum pressure up to 9.5×10^{-6} mbarwas used for sintering.

Table 1. Factor level (variables) in the sintering experiment

Factor		Level		
		0	1	2
A	Sintering Temperature (°C)	1200	1250	1300
B	Dwell Time (min)	60	120	180
C	Heating Rate (°C/min)	3	4	5
D	Cooling Time (min)	180	240	300

2. Results and discussion

Sintering process of samples was successfully accomplished and no cracks and deformation took place. Fig.3clearly compares a green part and a specimen after sintering. Basically, debinding leaves some open pores in components. Porosity is fraction of the component volume that is unoccupied by solid. Then, sintering process is normally associated by a considerable shrinkage. In other word, the pores are eliminated and the final dimension would be smaller than starting dimension. In this work, dimensional difference indicates 12.3% shrinkage aftersintering which is in the expected range for biomedical Ti-MIM products (12% to 15%).

(a)
(b)

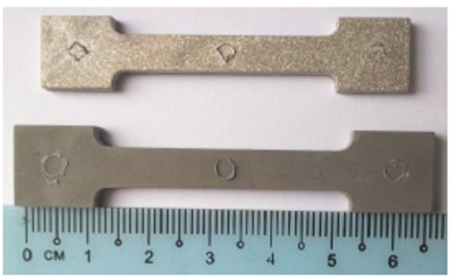

Fig. 3. A dimensional comparison between (a)sintered part and (b) greenpart.

Another objective of the experiments was to determine the effects of sintering factors on the sintered part and the optimum set of factors that would maximize the final strength. Basicallythe strength of the sintered part was measured by the tensile tests of the sintered parts using a servo-hydraulic structural test machine equipped with a 100 kN load cell at room temperature. A strain rate of 3.5 x 10^{-5} $s^{-1}$was applied. It carried out for three replications on each of the experimental condition (trials) as shown in Table 2.

Taguchi technique utilizes the signal noise ratio (S/N) approach to measure the quality characteristicdeviating from the desired value. It is also uses the S/N ratio approach instead of the average value toconvert the experimental results into a value for the evaluation characteristic in the optimum parameteranalysis [11]. Thestrength 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 Eq. (1):

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

whereY_{ij} is the amount of score for the green density and N is the total number of shots for each trial. The experimental results for the green density characteristic and corresponding S/N ratio using Eq. 1 is shown in Table 2 and the S/N response was plotted in Fig. 4. It is clearly shown that combination of A0, B0, C2, and D1 as the best set of factors. This means that the sintering temperature at 1200 ^{o}C; dwell time, 60 min; heating rate 5 ^{o}C/min and cooling time, 240 min the optimum levelthat could statistically result in maximization of strength.

Table 2. Orthogonal array and sintered strength

Trial	Factor				Measured Paremeter (MPa)			S/N
	A	B	C	D	Rep1	Rep2	Rep3	
1	0	0	0	0	780.528	764.406	872.251	58.0806
2	0	1	1	1	737.581	763.891	855.768	57.8539
3	0	2	2	2	811.311	913.344	899.401	58.8006
4	1	0	1	2	883.739	805.380	852.932	58.5421
5	1	1	2	0	834.903	773.501	725.774	57.7777
6	1	2	0	1	748.428	728.546	882.367	57.8219
7	2	0	2	1	692.639	643.306	773.990	56.8683
8	2	1	0	2	389.236	485.475	310.270	51.5015
9	2	2	1	0	188.973	239.878	528.610	47.8728
							Σ	505.1194
							▯	56.1244

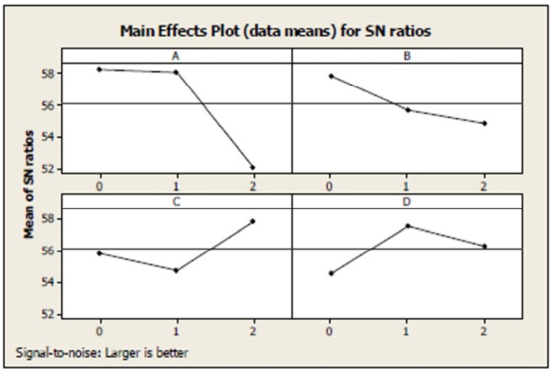

Fig. 4. The response plot of S/N Ratio

ANOVA has been generated to determine the statistical significance of the parameters as shown in Table 3 with at least 90% of confidence interval displayed 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 depicts a very significant level (α = 0.1) of each variables. Sintering temperature (A) is the most influential (21.3 %) to the sintered strength, followed by heating rate (C), the dwell time (B) and cooling rate (D).This result shows a good argument with Sidambe et al. (2009) which was found the sintering temperature to be a sensitive factor compared with others factors in determine the quality of Ti-6Al-4V sintered part in MIM process.

Hence the expected result at optimum performance is as shown in Table 4. The expected optimum performance is in the range of the optimum performance based on 90% confidence level which is $54.52<\mu<61.97$. The optimum parameter has been proven in the confirmation experiment that is conducted at the combined setting A0, B0, C2 and D1 and the result fell within the predicted 90% confidence interval as shown in Table 4. The optimum tensile strength can be achieved up to 934.3 MPa

Table 3.ANOVA for sintered strength

Variable	Factor	Degree of freedom	Sum of Squares	Variance	F	% Contribution
A	Sintering Temperature	2	24.5448	12.2724	2.8700*	21.2592
B	Dwell Time	2	4.7521	2.3760	0.5557	4.1160
C	Heating Rate	2	4.8361	2.4180	0.5655	4.1887
D	Cooling Time	2	4.3520	2.1760	0.5089	3.7694
	error	18	76.96978714			66.6667
	Total	26	115.4546807			100

* Significant at 90% significance level.

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

Significance optimum parameter: A0					
Optimum performance calculation: $\overline{T} + (\overline{A0} - \overline{T})$ 56.1243+(58.2451-56.1243) = 58.2451 dB					
Current grand average performance				:	56.1243
Confident interval (CI) at 90% confidence level				:	±3.7297
Expected result at optimum performance, μ				:	$54.5154 <\mu< 61.9748$
Confirmation experiment					
Repeat	1	2	3	Average	S/N ratio
Strength (MPa)	929	942	932	934.33	59.3672

From the analysis of the experimental results using the Taguchi method, sintered parts of Ti-6Al-4V with optimum parameter condition shows that the specimens have achieved the minimum requirement. Table 5 shows comparisonof sintered Ti-6Al-4Vwith ASTM B348-02 for the titanium alloy medical application. The oxygen, carbon and nitrogen contents will increase strength and decrease elongation. Control of these elements was the most importance issue in Ti-MIM. Although higher binder content and higher sintering parameter conditions were tested, no significant different in impurity levels was observed amongst the different configurations.

Table 5.Physical and Mechanical Properties ofSintered Titanium Alloy

Properties	Sintered Ti-6Al-4V	ASTM B348-02
Density (g/cm^3)	4.36 (97.75%)	>96%
Strength (MPa)	934.33	>895
Shrinkage (%)	12.27	12 to 15
Elongation (%)	12.14	>10
Oxygen (%)	0.00302	<0.2
Carbon (%)	0.056	<0.1
Nitrogen (%)	0.00004	<0.05

3. Conclusions

The use of binder palm stearin as a major friction in the MIM of Ti-6Al-4V has been successfully carried out. Sintered strength of the Ti-6Al-4V in MIM parts was optimized by using the Taguchi method. ANOVA showed that sintering temperature mostly affected the sintered strength. The optimum sintering parameter were found to be A0, B0, C2, and D1, corresponding to sintering temperature of 1200 °C, dwelling time of 60 minute, heating rate of 5 °C/minute and cooling time of 240 minutes. The experimental results shown that tensile strength of 934.33 MPa can be achieved when Ti-6Al-4V was sintered at the optimum condition. The physical and mechanical properties of sintered titanium alloy part were achieved the minimum requirement for sintered MIM parts in ASTM B348-02 for the titanium alloy medical application.

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