Parameter Optimization of Sintering Ti-6Al-7Nb Powder and Palm Stearin Binder System for the Highest Sintered Density using the Taguchi Method

Rozie Nani Ahmad
School of Manufacturing Engineering, Universiti Malaysia Perlis, Kampus Tetap Pauh Putra, Jalan Arau-Changlun, 02600 Arau, Perlis

Norhamidi Muhamad
Azizah Wahi
Phone: +60122740802; Fax: +6049885034
Department of Mechanical and Material Engineering, Faculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia.

Syarif Junaidi
Mechanical Engineering Department
College of Engineering
University of Sharjah, PO Box:27272, Sharjah, UAE, United Arab Emirates

ABSTRACT

The application of titanium (Ti) based biomedical materials such as Ti-6Al-7Nb is widely being used at present. Due to the toxicity of vanadium to the human body, the Ti-6Al-7Nb alloy has been developed as a more suitable biomaterial to replace the Ti-6Al-4V alloy. The sample was prepared by a metal injection moulding process, which is a net-shaped powder metallurgy forming process using palm stearin as a binder system. Meanwhile, the Taguchi method was used in conducting the experiment of the sintering process to investigate the effects of sintering factors on the density of the sintered parts. The density measurement was carried out by the Archimedes method using distilled water as a medium. The results showed that the sintering temperatures and dwell times were the most significant parameters that contribute to the highest sintered part density.

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Introduction

The Ti-6Al-7Nb alloy is currently being used for medical application, for example, as the artificial hip joints, spinal fixators, and dental implants, due to the lightweight and superior corrosion resistance. This alloy can be a good alternative to a more widely used Ti-6Al-4V alloy which is known to release vanadium ions that may cause long-term problems [1]. The α and β phases are bi-modal microstructures; a combination that exists in the alloy and is good for mechanical applications [2]. The application of Ti alloy as implant material usually required a complex fabrication process, high costs of the raw materials and geometrical design constraints [3]. The metal injection moulding (MIM) could be a smart choice to overcome these problems. One of the advantages of the MIM process is it suitable for mass production at a significant cost saving. Furthermore, MIM offers the unique design flexibility that is not readily achievable with other fabrication processes [4].

The traditional approach to the experimental work is to vary one factor at a time, holding all other factors fixed. However, this method does not produce a satisfactory result in a wide range of experiment settings because it can lead to wasting time and cost [5]. In this study, the Design of Experiment (DOE) method called the Taguchi method was adopted to determine and optimize the sintering parameters. In the recent years, the Taguchi method has become a powerful tool for improving productivity during research and development [6]. The Taguchi method was developed by Taguchi and Konishi [7]; the technique has been utilized widely in the engineering analysis to optimize the performance characteristics within the combination of design parameters [7]. The Taguchi technique is also a powerful tool for designing high-quality systems. It introduces a simple and efficient integrated approach in finding the best range of designs for quality, performance, and computational cost [7]. Toshiko and his friend also used the Taguchi method to find the effects of sintering conditions and improved their research in the mechanical properties such as tensile strength [8]. Furthermore, Nor Hafiez reported that the optimization of multiple sintering of Ti-6Al-4V compact quality performance using Taguchi method was successfully done at Universiti Kebangsaan Malaysia. The sintering parameters are very important during the sintering process [9]. The effect of four factors; sintering temperature, dwell time, heating rate, and cooling rate were investigated. The optimum sintering condition was proposed and confirmation experiments were conducted.

Methodology

Sample Preparation

The titanium niobium powder of average size 50 µm was purchased from MHC Industrial Co. Ltd (China) in irregular shape as shown in Figure 1. This powder was milled in a planetary ball mill (Fritsh pulverisette-6) to reduce the particle size to 24 µm. A hard metal ball and a bowl were used to prevent contamination and ethanol was used as the milling medium to avoid agglomeration and minimize temperature rise and oxidation. After the wet milling, the powder was dried in a vacuum oven at 100°C [10]. Figure 2 shows the image of the milled titanium niobium powder under the scanning electron microscope (SEM).

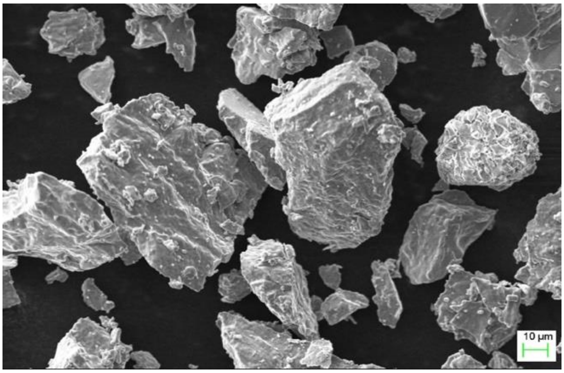

Figure 1: SEM of Ti-6Al-7Nb

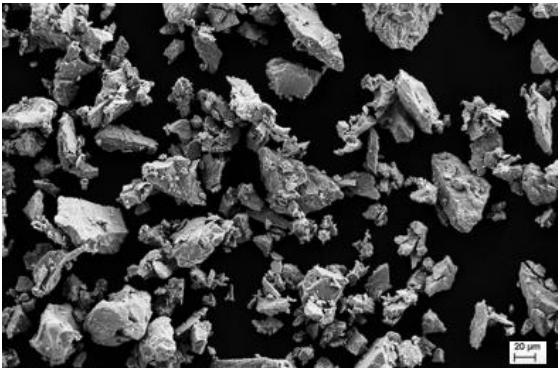

Figure 2: Ti-6Al-7Nb powder after ball mill scanned with an electron microscope

The major component of the binder used in the study was palm stearin supplied by Intercontinental Fats Sdn. Bhd. The back bone polymer was the low-density polyethylene (LDPE) purchased from Titan Chemicals Sdn. Bhd. The characteristics of the binder system are shown in Table 1.

Table 1: Binder Properties

Binder	Weight (%)	Melting Temperature (°C)	Density (gcm^{-3})
Palm Stearin (PS)	40	61	0.891
Polyethylene (PE)	60	127	0.950

The mixing process was then carried out to prepare the feedstock. With the homogenous feedstock, the injection moulding process was implemented. Lastly, the sintering process was carried out for these samples following the orthogonal array of the Taguchi method.

Design of Experiment

In this study, the L_9 (3^4) orthogonal array consisting nine (9) experiment trials and four (4) columns was used as the DOE followed by ANOVA to determine the significant level and contribution of each variable to the sintered part. The varied processing control parameters at three (3) levels for the sintering process are listed in Table 2. Figure 3 shows the flow chart for the simulation methodology.

Table 2: Control parameters and its value.

Level	Sintering Temperature (°C) A	Dwell Time (hour) B	Heating Rate (s) C	Cooling Rate (s) D
1	1200	3	3	4
2	1250	4	4	5
3	1300	5	5	6

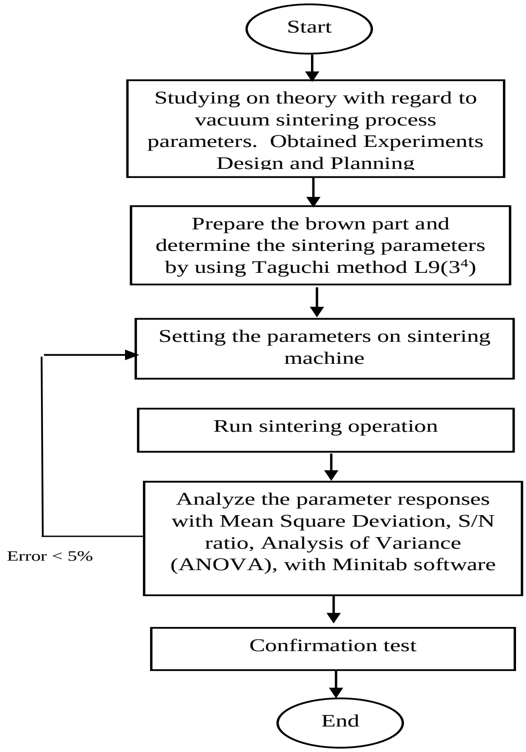

Figure 3: Flow chart of the methodology

Result and Discussion

The result of the experimental density analysis is shown in Table 3. A linear data pre-processing method for the density is the larger-the-better.

Table 3: Taguchi's L9(3^4) orthogonal array demonstrating the experimental value

Experiment	**Factor**				**Density (g/cm^3)**
	A	**B**	**C**	**D**	***S/N***
1	0	0	0	0	12.344
2	0	1	1	1	12.486
3	0	2	2	2	12.360
4	1	0	1	2	12.485

5	1	1	2	0	12.592
6	1	2	0	1	12.595
7	2	0	2	1	12.487
8	2	1	0	2	12.621
9	2	2	1	0	12.518
					∑112.49
					T12.50

Taguchi recommended analysing the means and S/N ratio using a conceptual approach that involves displaying the effects graphically and visually identifying the factors that appear to be significant, without using ANOVA, thus, making the analysis simple. Figure 4 shows the S/N ratio of each interaction where the optimum configuration becomes A2, B2, C1, and D2.

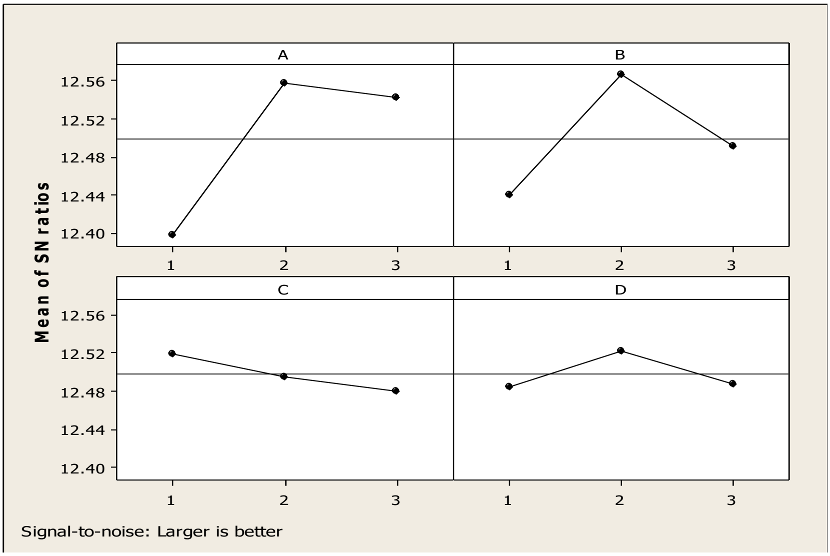

Figure 4: The Main Effect Plot for SN Ratio

Each of the parameters was analysed using the analysis of variance (ANOVA) which is a standard statistical technique to provide a measure of confidence. According to Table 4, the sintering temperature (Factor A) with 43.73% and dwell time (Factor B) with 21.53% contribution, are the highest significant parameters that contribute to the highest sintered part density.

Table 4: ANOVA Results

Factor	**Sum of Square**	**Degree of Freedom**	**Mean Square**	**Variance ratio F**	**Pure Sum of Square**	**Percent Contribution**
A	0.033	2	0.017	17.206	0.031	43.734
B	0.017	2	0.009	8.979	0.015	21.534
C	0.002	2	0.001	0.938	-0.000	-0.166
D	0.002	2	0.001	0.932	-0.000	-0.184
Error	0.017	18	0.001	1	0.025	35.082
Total	0.071	26	0.028			100

($F_{0.05;\,1;\,18}$)= 3.55(Table-F)
Confident Level: 95%

The expected result at an optimum performance is shown in Table 5. The optimum performance was as high as 4.2167 g/cm^3 while the range of the optimum performance based on 95% confidence level was $4.189 < \mu < 4.244$of the sintering density. The optimum parameter was proven in the confirmation experiment conducted at the combined setting of A2B2 and the result was within the predicted 95% confidence interval as shown in Table 6.

Table 5: Optimum sintering parameter and optimum performance

A2B2
Optimum performance calculation: $\overline{T}+ (\overline{A_2}- \overline{T}) + (\overline{B_2} - \overline{T})$ 4.2167+ (4.245-4.2167)+(4.249-4.2167) = 4.2773 g/cm^3
Current grand average performance : 4.2167
Confident interval (CI) at 95% confidence level : ±0.027494
Expected result at optimum performance , μ: $4.189 < \mu < 4.244$

Table 6: Confirmation experiment

Rep	**1**	**2**	**3**	**4**	**5**	**6**	**Mean (Larger the better)**
Parameter measurement	4.168	4.236	4.168	4.210	4.187	4.194	4.194

Figure 5 shows the fracture surface of the sintered sample. The corresponding elements of composition and distribution were investigated using the EDS mapping. The main elements inside the grain were titanium, niobium, and aluminium which were clearly distributed. Besides, it can be seen that the microstructure of the sintered part shows homogeneous distribution.

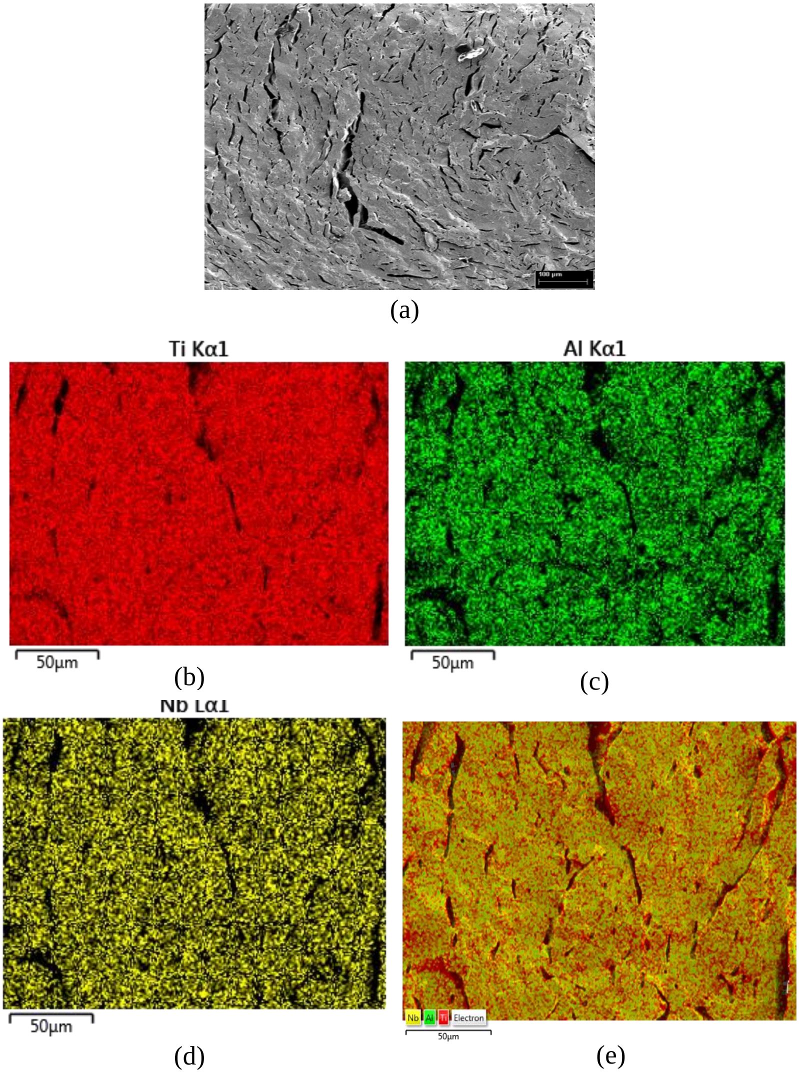

Figure 5 (a) SEM images of the fractured surface of the sintered part along with the corresponding EDS mapping of the constituent elements: (b) titanium (c) niobium (d) aluminium (e) combination of image (b), (c) and (d)

Conclusion

The Taguchi method was proven as an effective optimizing tool for a sintering process to produce the Ti-6Al-7Nb sintered part via the MIM method. In order to achieve the highest sintered density, the optimum combination of parameters achieved was A2, B2, C1, and D2 at sintering temperature 1250ºC, dwell time 4 hours, heating rate 3 seconds and cooling rate 4 second. The most influencing factor to the sintered density was the sintering temperature, followed by dwell time and heating rate while the cooling rate was the least significant and can be neglected. Furthermore, with this optimum combination, the homogenous sintered part will be achieved with the approval of the EDS result.

Acknowledgements

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