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Optimization of Compacting Process for Porous Ti-6Al-7Nb Alloys with Magnesium as a Space Holder by Using Taguchi Method

M N Fazlen, R. N. Ahmad*, N I N Suhaimi, M.S. Abdul Manan, and M. Marzuki

Faculty of Mechanical Engineering and Technology, Universiti Malaysia Perlis,
Kampus Tetap Pauh Putra, 02600 Arau, Perlis, Malaysia.

ABSTRACT

The optimization of compacting process for porous titanium niobium (Ti-6Al-7Nb) alloys with magnesium as a space holder prepared by powder metallurgy process was studied to analyze the significant porosity required in biomedical industry especially in bone tissue engineering. This approach helps to replace the damaged bones and solve the problem occurs during the implantation for artificial joints which is important that the implant material's stiffness is as similar to the joint bone as possible. The aim was to determine the significant factors affecting the physical and mechanical properties performance of porous Ti-6Al-7Nb alloys such as density and strength. Firstly, the Ti-6Al-7Nb were mixed with magnesium with weight percentage of 20%, 30%, and 40% as a spacer material for 30 minutes using Fritsch Pulverisette Mill mixer machine. Then, for compacting process, pressure in the range of 400MPa, 500 MPa and 600MPa were applied to the powder mixtures in a 13 mm cylindrical die by using manual hydraulic hand press machine. Compacting parameters have been optimized using Taguchi method of L-9 (3^4) orthogonal array. From the ANOVA results, density shows that holding time played as the most effected significant factor with 37.77% and compression was 59.47% for composition sample. The study demonstrated that many factors has been successfully optimized simultaneously, and a lot of quantitative information can be extracted from fewer experimental trials by using Taguchi method in compacting process.

Keywords: Magnesium, Porous, Taguchi, Titanium Niobium

1. INTRODUCTION

The key reasons why metals continue to be used in biomedical applications are because of its manufacturing, high strength and fracture resistance. With the combination of properties like low density, relatively low Young modulus, superior biocompatibility and corrosion resistance, titanium is typically preferred to other metallic biomaterials [1]. Titanium and some of its alloys are lightweight materials with the potential to withstand high temperatures. Because of their unusual combination of the properties, they are most widely used for various applications such as in the aerospace, automotive, and biomedical industries [2]. Meanwhile, titanium alloys had poor mechanical properties when it first used for surgical implants in comparison to other metallic materials such as Cr–Ni–Mo steel and Co alloys. However, increasing numbers of patients recording allergic reactions to Ni and Cr, elements found in austenitic steel used in osteosynthesis implants. As a result, most industries use titanium alloys to produce components used to connect broken bones, such as bone plates, bone screws, and intramedullary nails [3]. Therefore, in case of titanium-based materials, elemental titanium and the most popular titanium vanadium (Ti-6Al-4V) alloys were the first alloy to be introduced and used it for biomedical applications. However, due to problem possibilities about the impact of the dissolution in the chemical

* Corresponding author: rozie@unimap.edu.my

elements, in particular the arrangement of vanadium ions, and potential solutions for toxic effects, others alternatives have been suggested. The titanium niobium (Ti-6Al-7Nb) alloys was designed to replace the Ti-6Al-4V alloys because of its possible cytotoxicity and adverse reactions with the body tissues. Ti-6Al-7Nb alloys comes with similar mechanical performances to the Ti-6Al-4V alloy but better in biocompatibility [4].

Due to the arising of various applications of the biomaterials, porous titanium was chosen as a leading replacement for bone grafts and is a well-established bone implant material that makes improvement on bone ingrowth implant fixation combined with reduced risk of stress-shielding [5]. Cellular processes such as the migration and proliferation of osteoblasts and mesenchymal cells, as well as the transport of nutrients and oxygen which are needed for vascularization during the formation of bone tissue, can be facilitated by porous structures [6]. Therefore, the porous structure needs to be fully interconnected, has a precisely regulated pore size that can be optimized for cell attachment, proliferation, and migration, and has overall range of mechanical properties in terms of the bone. Moreover, the sufficient pore space allows for incorporation of hydrogels that release growth factors to increase the bone regeneration performance of the biomaterial maximally. Another essential characteristic of highly porous bone substitute is the surface area of the bone. It is understood that titanium alloys are bioinert in general and may be hydrophobic as well which by all means hydrophobicity may have a detrimental effect on cell attachment, and bioinertness means that the highly porous biomaterials' bioactivity ability remains unused. Bio-functionalization techniques may therefore be required to strengthen cell attachment and generate bioactivity to the surface of porous titanium bone substitutes. Since both surface plays an important role, biofunctionalization techniques could be targeted to achieve the best efficiency [7].

Various manufacturing methods have been developed involving the production of porous titanium. Among the methods of processing, powder metallurgy technology has been promising and given a maximum attention due to their remarkable advantages. By means of powder metallurgy techniques, the processing of titanium and its alloys is believed to be an appropriate way to reduce the manufacturing cost of titanium products where the master alloy addition version of the blending elemental method has been described as providing the cheapest way to produce titanium alloys with the desired composition [8]. A new powder metallurgy technique has been developed using space holder methods with the advantages of high uniformity, adjustable porosity, regulated pore shape and more uniform pore size distribution in recent years. Space-holder method is a developed powder metallurgy technology that enables porous structures to be created with regulated porosity and enhanced homogeneity [9]. This process works by the combination of the powder and the spacer, blend it and compacting the mixture before remove the spacer material, during or after sintering (depends on the type of the space holder use in the process) [10]. According to the previous study, magnesium was used as the space holder material due to its relatively low melting point in temperature that enabled the fast extraction of space holder during heat treatment. Moreover, magnesium vapor had limit the oxidation of titanium during sintering because of its highly affinitive to oxygen [11].

During the powder metallurgy process, compaction is the stage where the development of a green compact that can preserve the mixture and space containing particles throughout the following steps in the formation of the nanostructure. The process is where the mixture materials is pressed at high pressures to forms a green part, where the particles and nanostructure bond together by mechanical interlocking and cold welding. Pressing the materials at sufficiently high pressures will produce enough strength to be handled and machined. During the compact process, the amount of compression encountered by the powder will determine its green density and this in turn will control the amount of shrinkage or growth during the sintering process that the powder compact will undergo. It will also affect the final component's physical properties. By applying the pressure, particles of the mixture will rearrange, bond and the formation will harden the green compacts made of metal powders and spacer particles. After the part reach its maximum hardness, there is no density change with further application of compacting pressure. The compaction pressure ranges will depend on mixture and the tool material. The previous analysis recorded that the mixed powder of porous titanium alloy was compacted to steel cylindrical mold by applying pressure up to 800 MPa to obtain the desired shape with dimensions of the mold. It will cause a composite green part that consisting of matrix powder and spacer materials to be formed [12].

Yunhui Chen et al. [6] performed the uniaxially pressed at 600 MPa using a Carver 12 t Manual Hydraulic Press with a floating die to produce cylindrical shape of 10 mm in diameter and 5 mm in height by using magnesium as the space holder material. The calculated density is highly coherent with the designated value. The sample density is 2.25 g/cm^3, half of the amount of pure titanium (4.506 g/cm^3), when the porosity exceeds 50 vol%. Sergio Munoz et al. [13] performed compaction process by applying the pressure of 800 MPa using an Instron 5505 universal machine with the diameter of compaction die 8 mm. Mixing material which include titanium and ammonium bicarbonate as space holder, and total powder mass have been chose in order to produce samples in which to minimized the effect of compaction pressure. Hyoun-Ee Kim et al. [14] conducted the experiment by using magnesium as spacer where the mixture uniaxially cold-pressed into cylindrical green compacts at 600 MPa and 400 °C to obtain Ti/Mg compact. The temperature of the compaction and pressure value were chosen empirically. The results stated that the compacts obtained were not sufficiently strong below the temperature or pressure described, whereas by further increasing these values, almost no strength gain was achieved but Ti/Mg compacts show better machinability in conventional machining processes compared to the compacts using other spacer materials [15].

In order to minimize the experimental process, Taguchi suggested a specific design called orthogonal array to explore the entire parameter with a limited number of experimental trials. This approach can decrease the number of experiments and minimizes the effects of factors which cannot be monitored. In addition, it gives a simple, effective and systematic approach to fine the optimum cutting parameters in the process. Many researchers have applied the Taguchi method on powder metallurgy process to improve the quality issues and reduce cost from an engineering perspective. According to the experiment in [16], the mean value of parallelism data was reduced for two different products and decreased to 55.73% and 49.44%, respectively. The process used a L-8(2^6) experimental array with the aim to analyze several factors that can affect parallelism in the forming stage of the powder metallurgy process also to fine the significant parameter setting and verification. Initially, the full factorial method required for the experiment was 64 times, but only 8 experiments needed by applying the orthogonal array of the Taguchi method. Chao-Ton Su et al. [16] also used the Taguchi technique to enhance wire bonding quality parameters. The total cost saved was approximately USD 0.7 million, which indicated an improvement in production yield of 0.8 %. Soon, this Taguchi approach will be recognized to improve product quality which involve a simple data analysis and low experimental cost. Based on the study, this research will be focusing on optimization of the compaction process using Taguchi method during the fabrication of porous Ti-6Al-7Nb alloys using space holder method and magnesium as spacer materials.

2. DESIGN OF EXPERIMENT

Experiment can be conducted in many different ways to collect information and data from the results. Design of Experiment (DOE) is a statistical method used to perform experiment in a systematic way and get to analyze the data efficiently. This technique is statistical approach to reaction optimization that allows several variables to be varied simultaneously for a specific process. Moreover, this allows a large number of reaction parameters to be measured in a relatively small number of experiments. The main approach of DOE use here is Taguchi method.

3. EXPERIMENTAL DETAILS

3.1 Materials

Titanium niobium (Ti-6Al-7Nb) with spherical shape and particle size lower than 25μm was used as main material. The space holder material used in this experiment was magnesium with spherical shape powder (> 99.5%,) and particle size around 450μm and normally distributed size range of 250μm - 600μm. The composition of the mixing of Ti-6Al-7Nb with magnesium powders were determined with porosity of 20%, 30% and 40% that are aimed at the end of experiment.

3.2 Fabrication of Samples

By using powder metallurgy process, green compacts were produced from three types of mass required for the sample which is 1.5g, 2.5g and 3g. The weight percentage of magnesium were determined at 20%, 30%, and 40% with the size in the range of 250μm to 600μm from the overall mass of the sample while the rest composition was the Ti-6Al-7Nb alloys. Mixing process using Fritsch Pulverisette Mixer took 30 minutes with a speed of 100rpm thoroughly to ensure homogeneous distribution of the mixture. The compacting process was carried out by using a Hydraulic Hand Press machine together with a cylindrical double-ended die with an inner diameter of 13 mm using the pressure of 400MPa, 500MPa and 600MPa. This sample would be pressed for a specified holding time of 20s, 40s and 60s until the sample is in the form of a cylindrical.

3.3 Taguchi Method

In this regard, the compacting parameters examined by using Taguchi method of L-9(3^4) orthogonal array which decides that nine experiments will be conducted within three levels and four design factors (compacting parameters). Nine trials will be running for the compacting to find the optimum level of the process. Four parameters have been identified which is holding time, pressing value, composition sample, and weight. Table 1 and Table 2 shows the type of orthogonal array in Taguchi method that will be used to determine the significant factor. The Analysis of Variance (ANOVA) is the most widely used statistical treatment for the outcomes of experiments to assess the percentage contribution for each parameter. By measuring the mean square against an estimation of the experimental errors at specific confidence levels, ANOVA helps to formally assess the importance of all main variables and their interactions. Basically, ANOVA will be analyzing the results by using several formulas and obtain the percentage contribution of each control factors. Besides, Minitab 16 software also will be used to predict the optimum parameters and results.

Table 1 Level of various control factor

Factors	Level		
	1	2	3
Composition (%)	20	30	40
Pressing value (MPa)	400	500	600
Holding time (s)	20	40	60
Weight (g)	1.5	2.5	3.0

Table 2 L-9 (3^4) Orthogonal Array for Taguchi Method

Trials	Column of factor			
	1	2	3	4
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

4. RESULTS AND DISCUSSION

4.1 Density

This experiment is aimed to meet the best parameter for compaction of the porous Ti-6Al-7Nb alloys to achieve optimum density and compression results. The results were analyzed using MINITAB software, Mean Square Deviation (MSD), Signal-to-Noise (S/N) ratio, average factor effect, and Analysis of Variance (ANOVA). The density of the green specimen was measured according to Archimedes' Principle using Electronic Densimeter. The reading that corresponding to the replications were recorded for each experimental condition as shown in Table 3. According to the optimum condition of bigger is better (QC=B), the levels of the factors that contribute the highest average values are preferred [17].

From the response graph in Figure 1, the highest density obtained by the combination setting of composition sample at 20%, pressing value at 600MPa, holding time at 40 seconds and weight of the mixing material at 1.5g were the optimum level for compacting process. The best factors towards the S/N ratio also shows the same combination as shown in Figure 2. During the experiment, the highest densities obtained was 3.261g/cm^3 and the lowest densities obtained were 2.315g/cm^3. Healthy human bone mineral density (BMD) on average is around 3.88 g/cm^3 in males and 2.90 g/cm^3 in females [18] where density ranges of porous samples produced in study are consistent with the BMD range which can help to improve patient comfort and to keep rates of implant failure low. Based on Table 4 and Table 5, the ranking of the most influential factor is determined by optimum conditions the bigger the better [17] where the highest delta was pressing value, followed by holding time, composition sample and lastly weight.

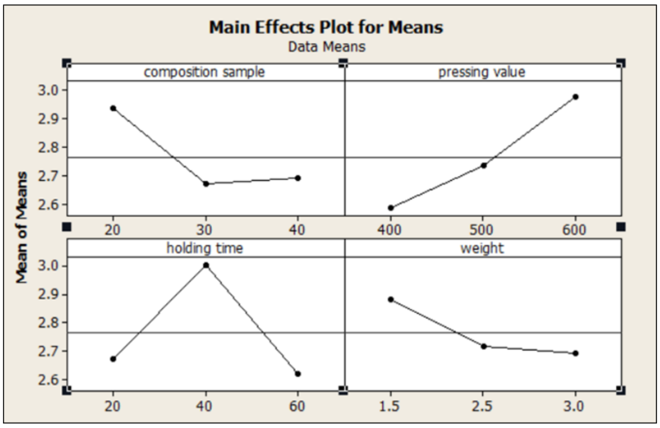

Figure 1. Main effects plot for mean of density

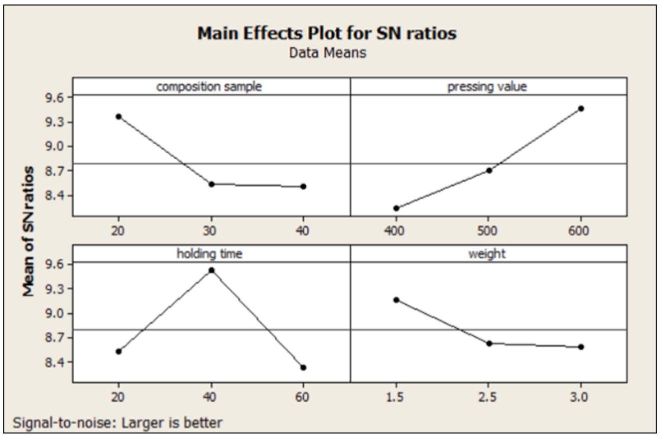

Figure 2. Main effects plot for Signal-to-Noise (S/N) ratio of density

Table 3 Density result of experiment layout

Exp.	Parameter				Density	MSD	S/N (dB)
	Composition sample (%)	Pressing value (MPa)	Holding time (s)	Weight (g)	(g/cm^3)	QC=B	
1	20	400	20	1.5	2.783	0.1291	8.8907
2	20	500	40	2.5	3.099	0.1041	9.8255
3	20	600	60	3	2.932	0.1163	9.3442
4	30	400	40	3	2.659	0.1414	8.4955
5	30	500	60	1.5	2.612	0.1466	8.3387
6	30	600	20	2.5	2.743	0.1329	8.7648
7	40	400	60	2.5	2.315	0.1866	7.2909
8	40	500	20	3	2.492	0.1610	7.9317
9	40	600	40	1.5	3.261	0.0940	10.2687

Table 4 Response table for mean from Minitab

Level	Composition sample (%)	Pressing value (MPa)	Holding time (s)	Weight (g)
1	2.938	2.586	2.673	2.885
2	2.671	2.734	3.006	2.719
3	2.689	2.979	2.620	2.694
Delta	0.267	0.393	0.387	0.191
Rank	3	1	2	4

Table 5 Response table for Signal-to-Noise (S/N) ratio from Minitab

Level	Composition sample (%)	Pressing value (MPa)	Holding time (s)	Weight (g)
1	9.353	8.225	8.529	9.166
2	8.533	8.698	9.529	8.627
3	8.496	9.458	8.325	8.590
Delta	0.856	1.233	1.204	0.576
Rank	3	1	2	4

4.2 Compression

The data obtained from the compression testing was the stress-strain diagram that shows the maximum stress and strain value as shown in Table 6. Young's modulus of the sample is calculated from two point of the straight line in the stress-strain diagram for each of the nine sample experimental trials. Mechanical properties like Young Modulus is very important to support transport of fluids during the implantation and should be similar to human bone [19]. Based on Figure 3, the highest mean for the composition sample was 40%, with a pressing pressure of 600 MPa, a holding time of 60 seconds, and a weight of 3g. Aside from that, the composition sample ranked first among the factors that impact the mean response, followed by holding time, weight, and finally pressing value as shown in Table 7.

The best factors towards the S/N ratio also shows the same combination as shown in Figure 4 and Table 8 where the ranking of the most influential factor is determined by difference value of delta whereas the highest delta was composition sample, followed by holding time, pressing value and lastly weight. Due to the homogenous distribution of powder particles which is a characteristic of the powder metallurgy method during mixing process, causing the higher level of interfacial bonding between matrix and reinforcement and which leads to better compressive strength [20].

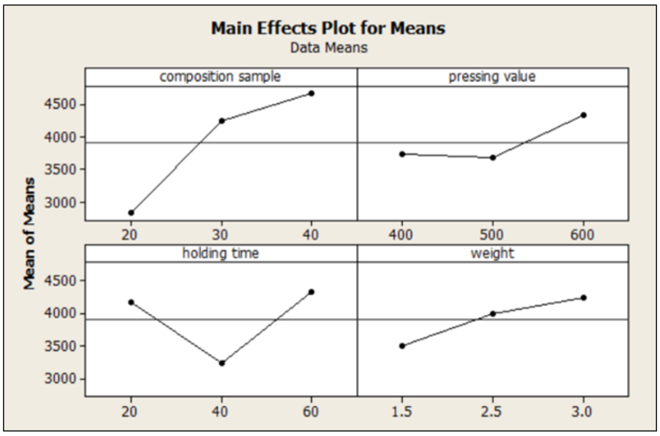

Figure 3. Main effects plot for mean of compression

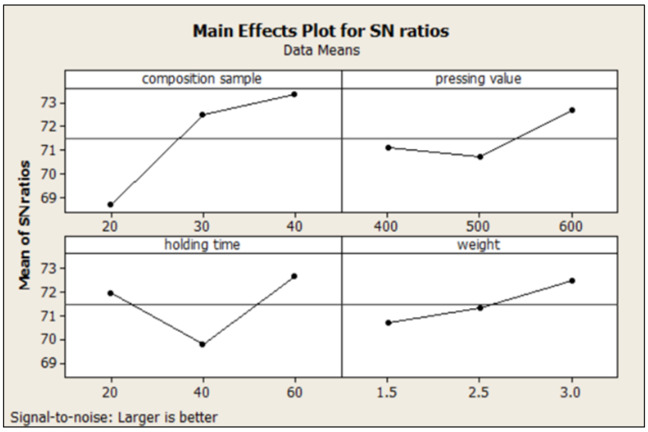

Figure 4. Main effects plot for Signal-to-Noise (S/N) ratio of compression

Table 6 Compression result of experiment layout

Exp.	Parameter				Young's modulus, E (MPa)	MSD	SNR
	A	B	C	D		QC=B	
1	20	400	20	1.5	2500	$1.6x10^{-7}$	67.9588
2	20	500	40	2.5	2000	$2.5x10^{-7}$	66.0206
3	20	600	60	3	4000	$6.25x10^{-8}$	72.0412
4	30	400	40	3	3704	$7.2888x10^{-8}$	71.3734
5	30	500	60	1.5	4000	$6.25x10^{-8}$	72.0412
6	30	600	20	2.5	5000	$4x10^{-8}$	73.9794
7	40	400	60	2.5	5000	$4x10^{-8}$	73.9794
8	40	500	20	3	5000	$4x10^{-8}$	73.9794
9	40	600	40	1.5	4000	$6.25x10^{-8}$	72.0412

Table 7 Response table for mean from Minitab

Level	Composition sample (%)	Pressing value (MPa)	Holding time (s)	Weight (g)
1	2833	3735	4167	3500
2	4235	3667	3235	4000
3	4667	4333	4333	4235
Delta	1833	667	1099	735
Rank	1	4	2	3

Table 8 Response table for S/N ratio from Minitab

Level	Composition sample (%)	Pressing value (MPa)	Holding time (s)	Weight (g)
1	68.67	71.10	71.97	70.68
2	72.46	70.68	69.81	71.33
3	73.33	72.69	72.69	72.46
Delta	4.66	2.01	2.88	1.78
Rank	1	3	2	4

4.3 Analysis of Variance (ANOVA)

ANOVA tests the relationship between categorical and numerical variable by testing the differences between two or more means. This test produces a p-value to determine whether the relationship is significant or not. For analysis the variance of results, there are several steps that need to be computed such as degree of freedom (DOF), sum of square, variance, F-ratio, and percentage contribution. Table 9 and Table 10 shows the ANOVA results for the density and compression affected the compaction parameters. For density, the factors that give most significant effect was holding time with the percentage contribution of 37.77%. It followed by pressing value at 30.98%, composition sample at 19.09%, and weight at 9.27%. Meanwhile, for compression, composition sample shows as the most significant factors which is 59.47%. Then, it followed by holding time at 22.70%, weight at 9.11%, and lastly pressing value at 8.71%.

Table 9 ANOVA for density

Factor	Composition sample (%)	Pressing value (MPa)	Holding time (s)	Weight (g)	Total
f_i	2	2	2	2	8
S_i	0.1332	0.2162	0.2636	0.0647	0.6777
V_i	0.0666	0.1081	0.1318	0.03235	0.33882
P_i(%)	19.0940	30.9787	37.7705	9.2707	100

Table 10 ANOVA for compression

Factor	Composition sample (%)	Pressing value (MPa)	Holding time (s)	Weight (g)	Total
f_i	2	2	2	2	8
S_i	5511470.2	807470.2	2103470.2	844803.53	9267214.13
V_i	2755735.1	403735.1	1051735.1	422401.8	4633607.1
P_i(%)	59.4728	8.7132	22.6980	9.1160	100

5. CONCLUSION

For density respond, confirmation experiment shows that all the prediction optimum compaction parameters were proved significantly from the result of percentage of error. The optimum compaction parameters were found to be corresponding to the composition sample of 20%, pressing value of 600MPa, holding time of 40 seconds, and weight of 1.5g based on mathematical analysis and Minitab software analysis. The percentage error between the new result of experiment and prediction results of density obtained was 3.6426% and 1.1881% which is not more than 10% of error. As for compression strength, the optimum compaction parameter was found with composition sample, 600MPa of pressing value, 60 seconds of holding time, and 3.0g of weight based on the predicted analysis result. Compared to these predicted results, it shows that the percentage of error was 8.8536% and 6.8759% respectively. ANOVA tables show that all the compaction parameters which are composition sample, pressing value, holding time, and weight affected the pressing process significantly. The ranking in the Taguchi method can be proved by the percentage of contribution in the ANOVA where for the density, holding time played as the most effected significant factor which was 37.77%, followed by pressing value at 30.98%, composition sample at 19.09%, and weight at 9.27%. And lastly for the compression strength, based on the ANOVA result, composition sample shows as the most significant factors which is 59.47%. Then, it followed by holding time at 22.70%, weight at 9.11%, and lastly pressing value at 8.71%.

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