

Influence Of Composition and Sintering Temperature on Density for Pure and Titanium Alloy Foams

S. Ahmad^a, N. Muhamad^b, A. Muchtar^b, J. Sahari^a, K. R. Jamaludin^c, M. H. I. Ibrahim^a, N. H. Mohamad Nor^b

^aFaculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, Batu Pahat, Johor, Malaysia

^bFaculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia, 43600 UKM Bangi, Selangor, Malaysia

^cCollege of Science and Technology, Universiti Teknologi Malaysia International Campus, 54100, K. Lumpur, Malaysia

*Corresponding author: sufizar@uthm.edu.my

Article history

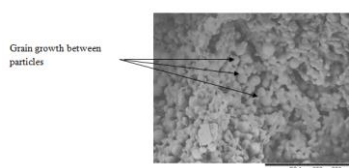
Received :29 July 2013

Received in revised form :

23 September 2013

Accepted :29 September 2013

Graphical abstract



Abstract

Metallic foams with high fraction of porosity have gained their usefulness and are now becoming a new class of materials for various engineering applications. Due to this, the present work aims to produce pure titanium and alloy titanium foams with high density using different composition and sintering temperature. The slurry method is selected to produce the pure titanium and alloy titanium foam. The titanium slurry is prepared by mixing pure or titanium alloy powder, polyethylene glycol (PEG), methylcellulose and water. Polyurethane (PU) foam is then impregnated in the slurry and dried at room temperature. This is later sintered in a high temperature vacuum furnace with different sintering temperatures. The density of the samples was tested using Archimedes test. From the result of analysis of variance, composition and sintering temperature affect the density of the samples for pure and titanium alloy foams. The suitable compositions of pure and alloy titanium are 60 wt%, 65 wt%, 70 wt% and sintering temperatures are 1200°C, 1250°C and 1300°C to produce a high density for the pure and titanium alloy foams.

Keywords: Slurry method; sintering process; PU; porosity

Abstrak

Logam berbusa merupakan satu bahan baru yang mempunyai sifat keliangan yang sangat baik dan sesuai untuk aplikasi dalam pelbagai bidang kejuruteraan. Disebabkan oleh sifat tersebut, kajian ini bertujuan menghasilkan titanium tulen dan aloi titanium berbusa dengan keliangan dan ketumpatan yang tinggi dengan menggunakan komposisi dan suhu pensinteran yang berbeza. Kaedah buburan telah dipilih untuk menghasilkan titanium tulen dan aloi titanium berbusa. Buburan titanium telah disediakan menggunakan campuran serbuk titanium tulen atau titanium aloi, polietilena glikol (PEG), metilselulosa dan air. Poliurethana (PU) berbusa direndamkan ke dalam buburan yang telah disediakan dan dikeringkan pada suhu bilik. Pensinteran di dalam relau vakum bersuhu tinggi dilakukan pada suhu pensinteran yang berbeza. Ujian ketumpatan yang dijalankan menggunakan prinsip Archimedes. Daripada keputusan analisis varian, kedua-dua faktor iaitu komposisi dan suhu pensinteran memberi sumbangan terhadap ketumpatan sampel yang dihasilkan. Komposisi titanium tulen dan aloi titanium yang sesuai digunakan untuk menghasilkan titanium tulen dan aloi titanium berbusa yang mempunyai nilai ketumpatan yang tinggi adalah 60 bt%, 65 bt%, 70 bt% dan suhu pensinteran yang sesuai adalah 1200°C, 1250°C dan 1300°C.

© 2014 Penerbit UTM Press. All rights reserved.

1.0 INTRODUCTION

Metal foams possess a unique combination of properties, such as air and water permeability, impact energy absorption capacity, unusual acoustic properties, low thermal conductivity, good electrical insulating properties and high stiffness with very low specific weight [1]. Because of these properties many researchers have been interested in producing good metal foams using various techniques. The basic processing techniques for

metal foams are liquid metallurgy, coating techniques and powder metallurgy [2]. In this study, the replication technique or slurry method has been chosen to produce metal foams.

This slurry method is included as a part of the powder metallurgy group because the starting material that was used is metal powder. Polyurethane foam templates are coated in slurry of metal powder followed by burning out of the template during the sintering process. This study is focused to produce a high density for pure and titanium alloy foams. Earlier works have

produced metal foams out of aluminum, zinc, lead, stainless steel and titanium. Overall, aluminum has been the most established metal for production in the form of foam [3]. Titanium and its alloy are excellent materials for lightweight applications at elevated temperatures and are widely used in aeronautical applications. Titanium foams have an additional potential for weight reduction and could even be suitable for functional applications if the pore structure were open [2].

This paper presents the results from research work on the effect of different sintering temperatures and composition of the pure and titanium alloy powder on density of pure and titanium alloy foam. The Taguchi method has been chosen to optimise the sintering and composition parameters of this study. Previous studies have shown that the important factors to produce good titanium foams are composition, sintering temperature, sintering time and heating rate [4-6]. The composition and sintering temperature factors have been used in this study. The effects of these factors were investigated and the optimum sintering condition was proposed using analysis of variance (ANOVA). From the analysis the suitable level of composition and temperature will be discussed and the most contribution factor will be found from this analysis.

2.0 EXPERIMENTAL

Initially, Polyethylene glycol (PEG) and methylcellulose (CMC) were stirred in deionised water for one hour. Pure titanium or titanium alloy powder was subsequently added to the solution and stirred for two hours. The composition that was used are 55 wt%, 60 wt%, 65 wt%, 70 wt% and 75 wt% of pure titanium and titanium alloy powder. The titanium slurry was used to impregnate polyurethane (PU) foam. The PU foams were dipped into the slurry and the dipping and drying processes were repeated until the struts of the foam were completely coated with pure titanium or alloy titanium slurry. The excess slurry was then removed by pressing the foam under a roller.

The samples were later dried in the oven for 24 hours at 30 °C. After the samples were completely dried, the PU was removed from the matrix by heating it at 600°C for 60 minutes. Subsequently, the samples were sintered at 1150°C, 1200°C, 1250°C, 1300°C and 1350°C with holding time of one hour. The rate for heating was 1°C/min. A liquid displacement method was used to measure the density of the samples after sintering.

3.0 RESULTS AND DISCUSSION

After sintering, the densities of samples were measured using Archimedes. The density will be increased followed by composition of the materials and lead to increase in the strength of the samples [7]. Figure 1 and Figure 2 shows the SEM micrograph of pure titanium and titanium alloy foams after sintered in vacuum furnace which consists of pores and struts. While, Table 1 and Table 2 shows the results of the analysis of variance (ANOVA) for sintering temperature and composition of pure titanium and titanium alloy foams. From the ANOVA for pure titanium foams, the composition of the sample offers significant at $\alpha = 0.005$ while for sintering temperature is only significant at $\alpha = 0.05$. The contribution for composition is 54.04 % and for sintering temperature is 6.54 %. The interaction between composition and sintering temperature did not give any significant level with a contribution to higher density only 1.04 %. In addition, ANOVA for titanium alloy foams shows both composition and sintering temperature factors are highly significant ($\alpha = 0.005$) for density. These variables contribute

about 63.34 % and 12.85 % respectively to the density. The interaction of this factor also did not give any contribution for the density.

Graph for the effect of composition of pure titanium and sintering temperature on the density of the samples are presented in Figure 3. While, Figure 4 shows the graph for composition and sintering temperature of titanium alloy foams. From these two graphs, the density was increased when the composition of titanium increases. From Figure 3 and 4, this trend was followed by the literatures [6&8]. It is because when sintering temperature increases, the necks will grow at the particle contact [9]. This phenomenon was shown in Figure 5 and Figure 6. Due to this, the pores will be eliminated and the density will be increased. The highest density for this research are 1.65 g cm^{-3} at 1350°C for pure titanium foam and 1.48 g cm^{-3} at 1350°C for titanium alloy foam. Other effects that can happen when the temperature was increased are greater shrinkage, grain growth, pore coarsening, less precision and higher properties [9&10].

From Surace *et al.* (2009) [11] the relative density will be increased when the composition of the sample was increased. We can see this trend as shown in Figure 3 and Figure 4. However, the lowest value for the density at 1150°C and 55% composition of pure titanium and titanium alloy foams. It is because the sample has more pores and the sintering temperature did not achieve the liquid phase sintering [9]. From Wen *et al.* (2002) [12] the result for density of the pure titanium foam was 0.90 g cm^{-3} using space holder method at 1200°C sintering temperature. However, for this research, the density at the same sintering temperature is 1.18 g cm^{-3} . So this research was getting higher density compared to the previous research. While for titanium alloy foam, the density result that was achieved was similar to the density result from other researcher [8].

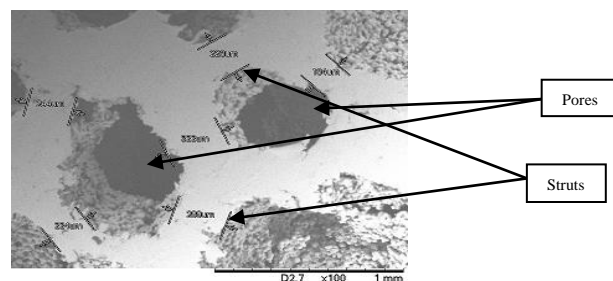


Figure 1 SEM micrographs of pure titanium foam sintered in the vacuum furnace

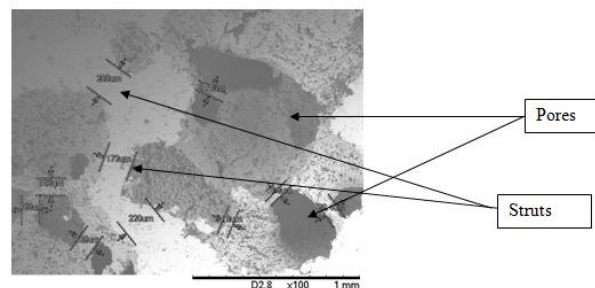


Figure 2 SEM micrographs of titanium alloy foam sintered in the vacuum furnace

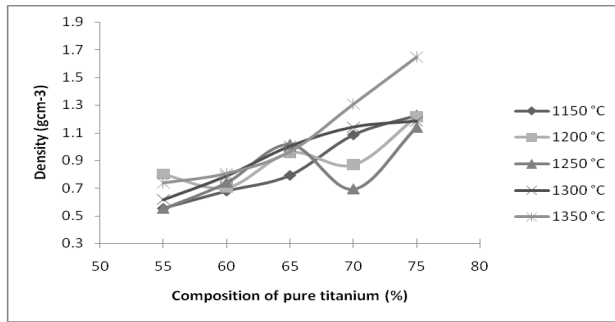


Figure 3 The effect of composition of pure titanium and sintering temperature on the density of the pure titanium foams

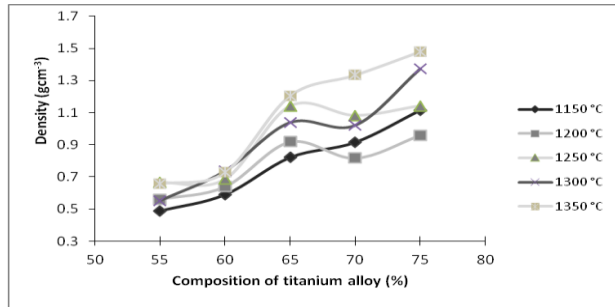


Figure 4 The effect of composition of titanium alloy and sintering temperature on the density of the titanium alloy foams

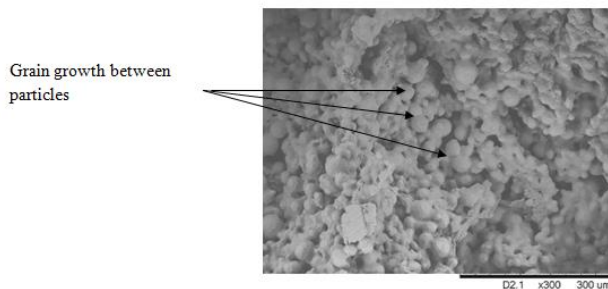


Figure 5 SEM micrographs of grain growth between particles after sintering process for pure titanium foams

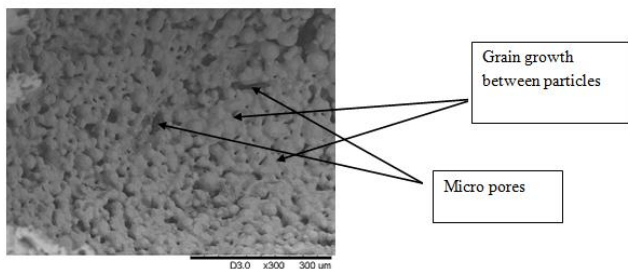


Figure 6 SEM micrographs of grain growth between particles after sintering process for titanium alloy foams

4.0 CONCLUSION

From the ANOVA analysis, the composition of titanium was found to give the most significant effect on the density of the samples followed by sintering temperature. Pure titanium and titanium alloy foams with high density have been manufactured successfully using the slurry method. The highest density for pure titanium foam is 1.65 gcm^{-3} for samples sintered at 1350°C . While, for titanium alloy foam the highest density is 1.48 gcm^{-3} at 1350°C sintering temperature. The suitable compositions of pure and alloy titanium are 60 wt%, 65 wt%, 70 wt% and sintering temperatures are 1200°C , 1250°C and 1300°C to produce a high density for the pure and titanium alloy foams. For further research, the compressive strength of these samples will be investigated.

Acknowledgement

The authors would like to thank the University Tun Hussein Onn Malaysia for their support in providing short term grant (STG-0787) for this project.

References

- [1] Derek, L. Steven, L. Jeffrey, A. R., Andrew, M, Donnamarie, A, Zach, S & Bryan, S. 2008. Mechanical Properties of Open-cell Titanium and Titanium Alloy Foams Made by PM Process. Proceeding of Advances in Powder Metallurgy & Particulate Materials. Washington, D.C.
- [2] Degischer, H. P. & Kriszt, B. 2002. *Handbook of Cellular Metals. Production, Processing, Application*. Wiley-VCH.
- [3] Ashby, M. F, Evans, A., Fleck, N. A., Gibson, L. J., Hutchison, J. W. & Wadley, H. N .G. 2000. *Metal Foams—A Design Guide*. United State: Butterworth-Heinemann.
- [4] Ryan, G. E., Pandit, A. S., & Apatsidis, D. P. 2008. Porous Titanium Scaffolds Fabricated Using a Rapid Prototyping and Powder Metallurgy Technique. *Journal of Biomaterials*. 29: 3625–3635.
- [5] Thieme, M., Wieters, K. P., Bergner, F., Scharnweber, D., Worch, H., Ndop, J., Kim, T. J. & Grill, W. 2001. Titanium Powder Sintering for Preparation of a Porous Functionally Graded Materials Destined for Orthopaedic Implants. *Journal of Materials Science: Materials in Medicine*. 12: 225–231.
- [6] Li, J.P., Li, S.H., Groot, K.de & Layrolle, P. 2002. Preparation and Characterization of Porous Titanium. *Key Engineering Materials*. 218–220: 51–54.
- [7] Ramay, H.R. & Zhang, M. 2003. Preparation of Porous Hydroxyapatite Scaffolds by Combination of the Gel-casting and Polymer Sponge Methods. *Journal of Biomaterials*. 24: 3293–3302.
- [8] Li, J. P., Li, S. H., Groot, K. de & Layrolle, P. 2003. Improvement of Porous Titanium with Thicker Struts. *Key Engineering Materials*. 240–242: 547–555.
- [9] German, R. M. 1996. *Sintering Theory and Practice*. USA: Wiley-Interscience Publication.
- [10] S. Ahmad , N. Muhamad, A. Muchtar, J. Sahari, K. R. Jamaludin, M. H. I. Ibrahim, N. H. Mohamad Nor and Mutadhahadi. 2010. Pencirian Titanium Berbusa yang Dihasilkan Pada Suhu Pensinteran yang Berbeza Menggunakan Kaedah Buburan. *Journal Sains Malaysiana*. 39(1): 77–82.
- [11] Surace, R., De Filippis, L. A. C., Ludovico, A. D. & Boghetich, G. 2009. *Journal of Materials and Design*. 30: 1878–1885.
- [12] Wen, C. E., Yamada, Y., Shimojima, K., Chino, Y. & Asahina, T. & Mubuchi, M. 2002. Novel Titanium Foam for Bone Tissue Engineering. *Journal of Materials Research*. 17(10): 2633–2639.

Table 1 ANOVA for sintering temperature and composition of pure titanium for density test after sintering process

Factor	Sum squared, S_n	Degrees of freedom, f_o	Variance, V_n	Variance ratio, F_n	Critical F value	Percentage of contribution, P_n
A(sintering temp)	0.63979	4	0.15994	7.228	$F_{0.005,4,25}=4.8351$	12.85
B (composition)	2.80518	4	0.70129	31.691	$F_{0.005,4,25}=4.8351$	63.34
A X B	0.29093	16	0.01818	0.822		0.00
Ralat	0.55323	25	0.02213			23.81
Total	4.28912	49				100.00

Table 2 ANOVA for sintering temperature and composition of titanium alloy for density test after sintering process

Factor	Sum squared, S_n	Degrees of freedom, f_o	Variance, V_n	Variance ratio, F_n	Critical F value	Percentage of contribution, P_n
A(sintering temp)	0.41240	4	0.10310	3.087	$F_{0.05,4,25}=2.7587$	6.54
B (composition)	2.43796	4	0.60949	18.249	$F_{0.005,4,25}=4.8351$	54.04
A X B	0.57852	16	0.03616	1.083		1.04
Ralat	0.83499	25	0.03340			38.38
Total	4.26386	49				100.00