



FABRICATION OF 316L STAINLESS STEEL FOAMS VIA POWDER METALLURGY TECHNIQUE

Z. Abdullah, S. Ahmad, M. F. M. Rafter and N. S. A. Manaf

Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia Parit Raja, Batu Pahat, Johor, Malaysia

E-Mail: sufizar@uthm.edu.my

ABSTRACT

Metal foams can be classified as lightweight materials which are low densities, having a unique combination of physical and mechanical properties, energy absorption and good thermal conductivity. 316L Stainless Steel has widely used by researchers as material to fabricate metal foams due to its high-strength-to weight ratio, biocompatibility and corrosion resistance and successfully used in implant applications. The major challenges that need to be focused while producing metal foam is the mismatch of the properties between bones and the metallic material. Due to this mechanical mismatch, bone is insufficiently loaded and become stress shielded, which eventually leads to bone resorption. Thus, there are factors need to be considered includes the interconnecting pores that suitable with bone, the pores, shape and density of the implants same with the pores, shape and density of the bone. This research is to fabricate the 316L Stainless Steel (SS316L) foam prepared by Compaction technique and to study and characterize the properties of SS316L foam after sintering process. The SS316L have used as a raw material and Polyethylene glycol (PEG) and Carbamide are used as a binder and space holder respectively. The material was mixed by using ball milling machine to get the homogenous mixture. After that the compaction process was held by using conventional axial pressing. This process is known as powder metallurgy technique. The Properties Characterization was measured by doing density and porosity test, Thermal Gravimetric Analysis (TGA), and Scanning Electron Microscopy (SEM).

Keywords: porous metals, pressing method, physical properties.

INTRODUCTION

At present, high requirement of lightweight constituent make metal foams extremely attractive as an industrial technology for biomedical application which is demanding on weight reduction [1]. The physicality of metal foam is high porosity make metal foams very lightweight. Basically, metal foams are artificial porous medium that has solid matrix structure of metal consist of empty or fluid-filled voids. Metal foams have been characterized into two types which are open-cell and closed-cell. The foams is called open-cell when the voids are connected via open pores, but when the foams are separated by the solid walls and not connected via open channel are described as closed-cell [2].

In order to achieve the metal foams which suitable in orthopedic application and have good properties such as low density, high strength-to-weight ratio, excellent mechanical properties, biocompatibility and corrosion resistance, the suitable materials have to choose carefully. Metals are the most suitable material to fabricate the metals foam proportionate to the ceramic and polymer. Even though the ceramic material have excellent corrosion resistance but ceramic cannot being employed as load bearing implants due to their brittle properties, whereas polymeric systems cannot sustain the mechanical forces present in joint replacement surgery [4]

There are various types of metal that have been used as main materials to fabricated metal foams includes titanium, titanium alloys, nickel, aluminum, magnesium, and stainless steel [3]. Since early 1960s, Stainless steel widely used in orthopedic application such as fabrication of femoral stems, balls and acetabular cups, fabrication of

knee and femoral components and tibial trays because of its biocompatibility and inexpensive [5].

Over the years in line with the development of technology, there are variety of fabrication process have been developed due to produce metal foams. In order to produce porous metallic foam, there are some techniques which suitable to apply such as slurry foaming, extrusion of polymer or metal mixtures, and space holder method [6].

There are large varieties of fabrication techniques for metal foams or similar porous structures but usually favorable technique is liquid phase or powder metallurgy process. Previous researcher stated that metal foams was successfully fabricated via Powder Metallurgy technique. By using compaction method, metal powders are mixed with foaming agent and then compacted by using hot pressing, cold pressing, hot extrusion, or co-extrusion. The final product of the compaction process is a dense foamable material that can be worked into sheets and profiles [8].

This research is to fabricate the 316L Stainless Steel (SS316L) foam prepared by Compaction technique and to study and characterize the properties of SS316L foam after two-stage sintering process. As a result, the formation of pore structure and the physical properties of the metal foams were studied.

EXPERIMENTAL PROCEDURES

In this research, the raw material that involved in order to produced metallic foams are type 316L Stainless Steel as the metallic material, Carbamide as the space holder material and Polyethylene Glycol (PEG) as the binder. The SS316L particles is spherical shapes while



both PEG and Carbamide are water soluble materials. The particle size of the Carbamide is less than 200 μm . The SS316L powders, PEG and carbamide were mixed homogenously by using ball milling machine at 60rpm for about 10minutes. The samples were produced by pressed the mixture at 3 tonnes by using hydraulic press machine. Then, the last stage involved was heat treatment was applied to the samples in box furnace in two stages. For the first stage represents for decompose of the carbamide particles at temperature of 280 $^{\circ}\text{C}$ for 30 minutes, and the second stage at 870 $^{\circ}\text{C}$ for 1 hour to sinter the SS316L metal. The temperature of 870 $^{\circ}\text{C}$ was defined as the maximum temperature of services temperatures in air for the SS316L stainless steel [7].

Thermogravimetric analysis (TGA) TGA was performed in order to know the high temperature for space holder material Carbamide and can be applied the temperature during sintering process. In this research, the density and porosity is measured by using the Archimedes Principles for the samples after sintering process. The microstructure of the metal foam was observed by performed the Scanning Electron Microscopy examination. In this study, the microscope examination of the metal foam was done after sintering process to determine the formation of pores from the cross section of the samples, characterize the pore structure for the samples, the densification of the samples and the development of neck growth of the samples structure.

RESULT AND DISCUSSIONS

These sections discuss about the result of the SS316L foams after finish the sintering process and after all the testing have been conducted. The TGA was done to obtain the suitable value of temperature for Carbamide in order to ensure that Carbamide will fully decompose during sintering process from the TGA result, the temperature value for eliminate the Carbamide particles at first stage of sintering process can be decided as 280 $^{\circ}\text{C}$ which mean nearly the temperature of second phase. There was previous study of TGA analysis which gets the result of 350.75 $^{\circ}\text{C}$ different value from this study, it because of the different of the Carbamide type [9]. The temperature for the second stage of sintering process is 870 $^{\circ}\text{C}$. The temperature value of 870 $^{\circ}\text{C}$ represent as the maximum oxidation services temperature in air for the stainless steel type 316L which can be categorized as heat resisting steels. The consideration of the value of the temperature to sinter the SS316L foams is important due to the use of box furnace for the sintering purpose which influences the samples condition after finished sintered.

Density and Porosity test has been carried out to determine the bulk density and the percentage of porosity after the sample finish sintered. This is because of the contamination occurred to the sample during the density test. From the result obtained, it shows that the value of the bulk density will directly proportional with the increasing of the weight percentages (wt. %) of SS316L composition added up to the samples. The increasing value of the density happen due to the densification process is occurring at this stage [10]. It also can be supported by

the previous researcher which stated that the density is proportional to the amount of the 316L particles in the powder formulation [1]. The porosity test of the samples show the difference result compare to the density result. When the amount of the 316L powders was increased in the powder formulation, the value of the porosity was decreased. This is caused by the neck grows at the particle contact that causes the elimination of pores and increase in density during sintering process [10]. This result can be supported by previous researcher that obtains the decreasing of the porosity value with the increasing of the amount of SS316L particles.

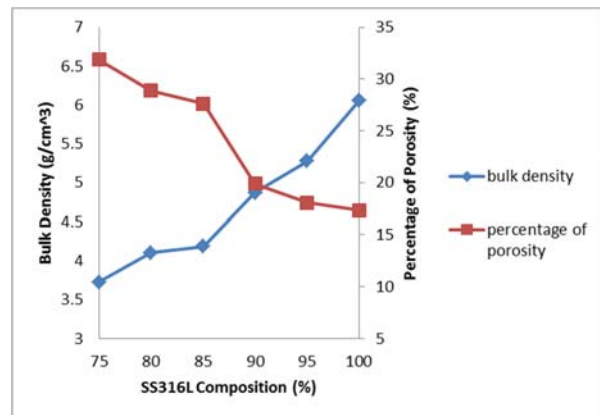
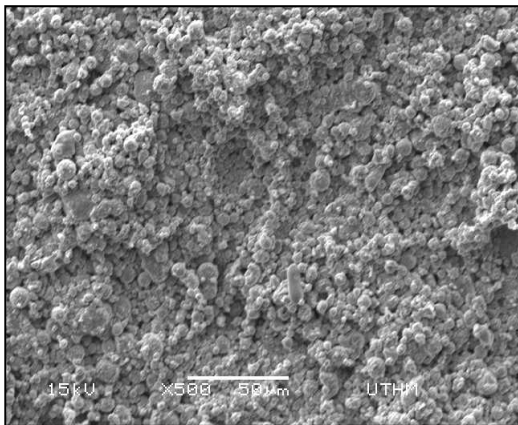
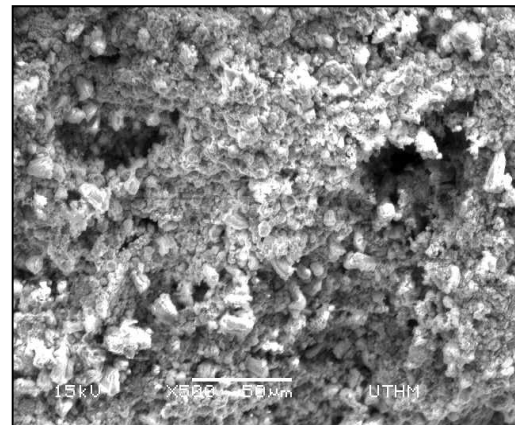


Figure-1. Result of density and porosity test of SS316L foam fabricated by powder metallurgy techniques.

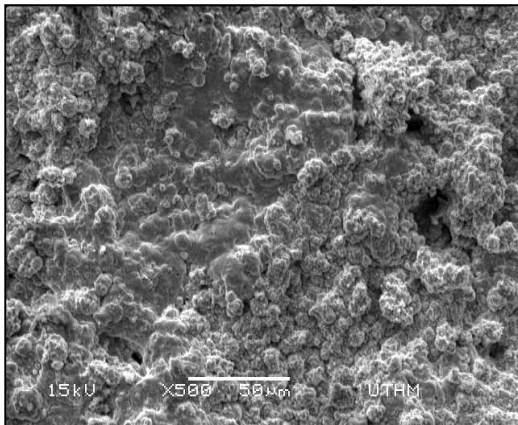
Scanning Electron Microscopy (SEM) analysis was done to study the microstructure and the morphology of the SS316L foams. In addition, the analysis also discusses the structure and pore distribution and the inclusion of the particles of the SS316L foams. Figure-2 shows the comparative set of morphology for the SS316L foams after sintering process. From the Figure-2(a) which represent the morphology of the pure SS316L foam, even though the temperature for the sintering process is 870 $^{\circ}\text{C}$, it can be seen that the grains is started to growth and consolidate between particles and densification occurred. Figure-2(b) show the morphology for SS316L foam with synthesized of 5wt. % of Carbamide, which the pore started to form. The pore distribution can be classify as heterogenous and consist with the irregular pores but from the morphology, the densification of SS316L foam can be seen clearly. Mariotto et. al in her study to fabricate SS316L also obtain the same pore distribution which the pore distribution is very heterogenous [11].



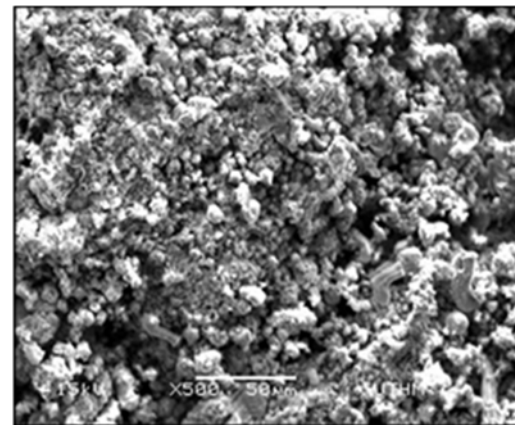
(a)



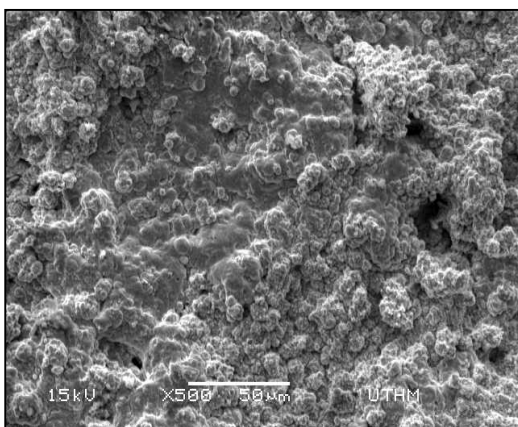
(d)



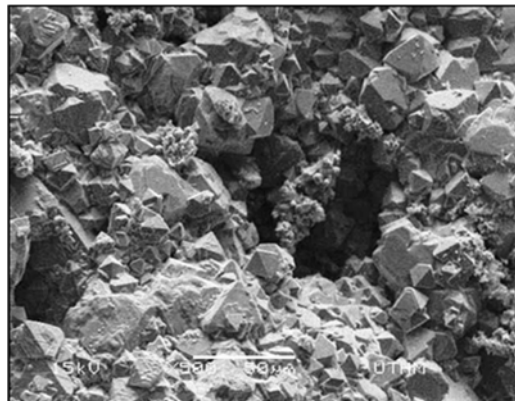
(b)



(e)



(c)



(f)

Figure-2. SEM micrographs of 316L stainless steel foams: a) pure SS316L b) 5 wt. % Carbamide c) 10 wt. % Carbamide d) 15 wt. % Carbamide e) 20 wt. % Carbamide; and f) 25 wt. % Carbamide.

Figure-2(c) and Figure-2(d) represent the morphology of the SS316L foam with added up 10wt. % and 15wt. % of Carbamide respectively into the solution. From the morphology, inhomogenously pore structure was obtained due to the different shape and size of the pore while the pore distribution show that the pore is clustered



which can be explained that the pore was gather between each other and isolated from the other pore. The inclusion of the SS316L particles clearly visible which the particles consolidate between each other. The morphology of the SS316L foam with the addition of 20 wt.% Carbamide can be seen in the Figure-2(e) which contains the pore which can be categorize as the micro pores. Other than that, it has been observed that, the samples contain fibrous structure which these structures indicate the oxidation present to the samples.

Figure-3 shows clearly the structure of the fibrous structure with the magnification of 1500X for the SS316L foam with 20 wt. % Carbamide. The pore distribution for the SS316L foams with 20 wt. % Carbamide is not uniform. It can be seen that the formation of pore is dispersed each other. Other than that, Figure-2(f) show the samples with the composition of 20 wt. % of Carbamide particles shows the less pore distribution with the large pore size. The structure of this sample can be classified as having side or cubic near to tetragonal structure. Figure-4 shows clearly the structure of the cubic near to tetragonal structure with the magnification of 1500X for the SS316L foam with 25 wt. % Carbamide. It can be conclude that the formation of this structure may be cause by the chemical reaction due to oxidation. Chemically, if the oxidation number of metal is high, the metal can accept more electrons which are Metal cat ions [11].

CONCLUSIONS

The SS316L metal foams had been successfully fabricated via powder metallurgy technique yet there are some improvements have to be done due to increase the strength of SS316L metal foam in order to proceed to mechanical testing. From the result obtained, the metal foams with the 75 wt% of SS316L produce more pores compared to the other composition with the lower value of density and higher value of percentage of porosity make it suitable for the implant application. Overall, the result show that the metal foams had micro pore which can be categorized as closed pore and there are particles that well growth and combine with each other and develops sinter neck growth.

There are some recommendations and suggestion that might be help further to improve the research. Reduce the amount of 316L Stainless Steel powders to the composition of the mixture and add the amount of Carbamide in order to produce more pores. Other than that, the sizes of particles of Carbamide which are needed to reduce by the crushing process have to control in order to avoid the particles of the Carbamide powders become too small. This is because, if the particles sizes of Carbamide powders are too small, the larger pores of the foams can't be achieved. In addition, during compaction process, the pressure applied has to be increase to produce the green body which has high strength and strong enough to carried to the sintering stage. The sintering process also has to be done by using vacuum furnace to avoid oxidation.

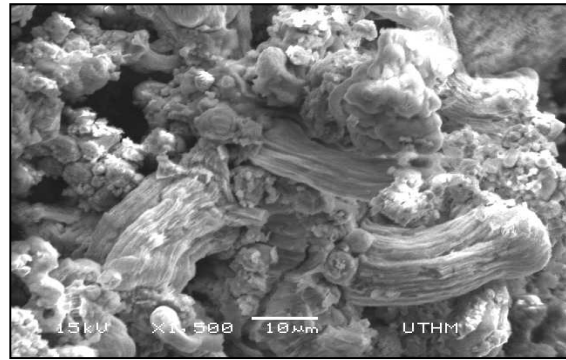


Figure-3. The structure of the fibrous structure with the magnification of 1500X for the SS316L foam with 20 wt. % carbamide.

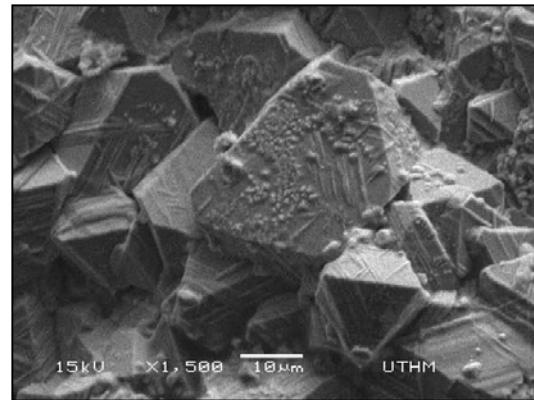


Figure-4. The structure of the cubic near to tetragonal structure with the magnification of 1500X for the SS316L Foam with 20 wt. % carbamide.

ACKNOWLEDGEMENT

Authors would like to acknowledge the support from Malaysian Ministry of Education for the financial support through Exploration Research Grant Scheme (ERGS-E028).

REFERENCES

- [1] Gauthier, M. Structure and Properties of Open-Cell 316L Stainless Steel Foams Produced by a Powder Metallurgy-Based Process. MetFoam'2007, 5th International Conference on Porous Metals and Metallic Foams. 5-7 September 2007. Lancaster, Pennsylvania: DEStech Publication, Inc. 2007. ms 149-152.
- [2] Dukhan, N. Metal Foams: Fundamentals and Applications. Lancaster, Pennsylvania: DEStech Publication, Inc. 2013.
- [3] Rosip, N. I. M., Ahmad, S., Jamaludin, K. R. and Noor, F. M. 2013. International Conference on Mechanical Engineering Research (ICMER2013), 1-3



July 2013 Bukit Gambang Resort City, Kuantan, Pahang, Malaysia Organized by Faculty of Mechanical Engineering, Universiti Malaysia Pahang Paper ID: P138, (July), 1-3.

- [4] Ryan, G., Pandit, A. and Apatsidis, D. P. 2006. Fabrication methods of porous metals for use in orthopaedic applications. *Biomaterials*, 27, 2651-2670.
- [5] Davis, J. R. Handbook of Material for Medical Devices. Materials Park, Ohio: ASM International. 2003.
- [6] Banhart, J. 2001. Manufacture, characterisation and application of cellular metals and metal foams. *Progress in Materials Science*, 46, 559-632.
- [7] BSSA. 2015. Maximum service temperatures in air for stainless steels. <http://www.bssa.org.uk/>.
- [8] Banhart, J. and Baumeister, J. 1998. Deformation characteristics of metal foams. *J. Mater. Sci.* 33, 1431.
- [9] Rafat, F. 2014. Effect of different heating rate on the thermal decomposition of urea in an open reaction vessel, 6(5), 75-78.
- [10] Ahmad, S., Muhamad, N., Mughtar, a, Sahari, J. and Jamaludin, K. R. 2010. Development and Characterization of Titanium Alloy Foams, 5(2), 244-250.
- [11] Mariotto, S. D. F. F., Guido, V., Yao Cho, L., Soares, C. P., and Cardoso, K. R. 2011. Porous stainless steel for biomedical applications. *Materials Research*, 14(2): 146-154.
- [12] Volicer, B. J. and Tello, S. F. 1998. D-Metal Complexes. UMass Lowell.