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The Effect of Different Urea Composition on Production of Porous Stainless Steel type 316L through Powder Metallurgy Technique

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ABSTRACT. Recently, there has been renewed interest in fabrication of porous metals and metallic foams as biomedical implants. Metal foams have great properties such high stiffness, great mechanical strength and high energy absorption that cannot be obtained from ceramic and polymer foams. The present study aims to fabricate the porous stainless steel by using urea as space holder material through powder metallurgy technique. Stainless steel (SS316L) powders was used as metallic material were mixed with urea and polyethylene glycol (PEG) as binder using ball milling machine and then compacted at 8 tons. The composition of urea particles into the formulation are 45 wt.%, 50 wt.%, 55 wt.%, and 60 wt.%. The two-stages sintering process was performed which at the first stage, the temperature applied was 400 °C for 2 hours to remove the urea particle in the green compacts and the second temperature at 1100 °C for 2 hours to sinter the steel. The characterization of the samples after sintering process was carried out by performing density and porosity test and scanning electron microscopy (SEM) was conducted to identify the morphology characteristic. The findings show that, the sample with 40 wt.% of urea composition produced high density value of about 4.36 g/cm^3. The sample with 60 wt.% of urea composition produced larger pores in their structure and obtained high porosities value of about 56.22%.

KEYWORDS: *Cellular metals, Urea, Space-holder technique, Sintering;*
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1. INTRODUCTION

The combination properties of metals which are tough and thermally and electrically conductive and porous which synonym to lightweight behaviour will produce the unique characteristics of porous metals [1,3]. Porous metals can be classified into closed-cell and open-cell foams. In closed-cell foams, the pores was separated by a metal thin wall, whilst in open-cell foams the pores was interconnected in each metallic strut frames [3,4]. Porous metals have gained the attention from researchers due to their interesting properties such low densities, high bending strength, thermal and electrical conductivity and great acoustic properties [5]. Porous metals are well known as lightweight structures that usually applied in aerospace and automotive industries, as functional application such filters, electrode, catalyst, as well as dental and orthopaedic devices in biomedical application [6]. Porous metals with adjustable density, high porosity and excellent combination of properties have great potential for biomedical application as bone implant. However, there is major challenging concerning porous metals as bone implant. Mismatch of Young's moduli between implant and the surrounding bone lead to implant loosening and may cause stress shielding of bone [6]. In order to overcome these problems, it is important to form highly porous implants which can provide better biological fixation and enhanced bone in growth into the pores network of the implants [7].

Numerous research has been successfully used metals as bio implant for many years and cobalt-chrome based alloys and stainless steel are the first metals that successfully used in orthopaedic application during the twentieth century [8]. Stainless steel in one of the principal metals that usually choose for structural application such as orthopaedic implants and shoulder due to properties like ability to bear significant loads, high fracture toughness, biocompatibility and high corrosion resistance [3,10].

Numerous fabrication methods have been studied in order to produce and develop porous metals efficiently. Solid state methods via powder metallurgy technique based on space holder method are the most promising method for manufacturing porous metals. The advantage of this technique is the process variables can be easily control, the pore size and the structures can be designed, and the fabrication process can be performed at lower temperature thereby decrease the severe chemical reactions [10]. By performing this technique, the shape, size and volume fraction of porosity can be controlled which the range of porosity in about 60-80% can be formed. The space holder method starts by mixing the metallic powder along with space holder materials and binder homogenously. Then, the mixtures were compacted using uniaxial pressing machine and the resulting product called as green compact. Heat treatment was apply to the green compact at relatively low temperature in order to remove the space holder material from the compact, and the next stage the compact is sintered at high temperature [11].

This study set out to produce the porous stainless steel type 316L foams by using compaction method with different composition of urea particles as space holder materials. The green compact was sintered in two-stages sintering process using tube furnace under high purity Argon atmosphere. The characterization of the sintered porous SS316L was analyzed by performing density and porosity testing and the morphology analysis of the foams was evaluated by using SEM apparatus. The influence of the composition of urea on the formation of stainless steel foam and the pore distribution was studied.

2. MATERIALS AND METHODS

The sintered powder compacts were prepared using stainless steel type 316L powder with the particle size of 7.157 µm (D50) purchased from Maju Scientific Sdn. Bhd. The spherical SS316L powders with the composition of 40, 45, 50, 55, and 60 wt.% was mixed with urea with the particle size of 200 µm and 1 wt.% of PEG by using ball milling machine at 60 rpm for about 15 min. The samples of 14 mm diameter and 26 mm thickness in size were compacted in cylindrical die by using Carver pressing machine at a pressure of 8 tons. Urea particles was removed in a tube furnace at a temperature of 400 °C for 2 hours and then was sintered at temperature of 1100 °C for 2 hours with heating and cooling rate of 2 °C/min in high purity of argon (Ar) atmosphere with flow rate was maintained at 51 ml/min. The physical properties of the samples was analyzed by performing density and porosity test by applying Archimedes' principle and morphology analysis was analyzed by using JEOL scanning electron microcopy (SEM).

3. RESULTS AND DISCUSSION

The microstructural analysis was conducted to determine the pore size and strut produced. The pores and struts inside the porous structure of porous SS316L for 60, 55, 50, 45, and 40 wt.% of the urea samples were observed by SEM. The sizes of the pores are in the range 169.99 µm to 198.90 µm and can be categorized as large size pores (50-400 µm) [8,9]. Fig. 1 shows an overview of pores and slag generated from the samples of all compositions with the sintering temperatures of 1100 °C for the sintered samples performed in argon environment by using the tube furnace. All samples have clearly produced pores and struts.

Fig. 1 (a) presents the sample with 60 wt.% of urea content has open pores and well-developed of struts but the surface was too rough. This may be due to the amount of the space holder was too excessive than SS316L powder, causing it to become unable to coat the grain particle of space holder effectively. In fact, it is very tough to control the process in different metal and space holder powders [14].

In addition, it is difficult to control the cell size and its distribution practically especially for micro porous metal [15]. As shown in Fig. 1. (c,d), for 55 wt.% of urea content, non-uniform pore distribution was formed on the sample structure. This is caused by the irregular shape and non-uniform size of the space holder particles [12]. As shown in Fig. 1 (e,f), the pore distribution of the sample with 50 wt.% of urea content can be categorized as clustered and the cluster pores are apart from each other. Fig. 1 (g,h) displays the SEM image for stainless steel foam with composition of 45 wt.% of urea content. It can be seen from the Fig. 1 (h) the formation of large pore is less likely to occur and small pores was formed in the sample structures. It is possible that the decreasing the composition of urea into the formulation may reduce the formation of large pores. Fig. 1 (i,j) displays the structure of the sample with 40 wt.% of urea content. The closed pores were generated and a thicker structure of struts increased comparing to other compositions. It is proven by the average of the bulk density in this composition is higher compared to other. The details of bulk density of the samples are as shown in Fig. 2.

Fig. 2 presents the graph of sintered density and apparent porosity versus compositions of SS316L powders. The highest density value at the sample with 40 wt.% of urea content was recorded. The result shows that the sintered density of the porous SS316L increase with the decreasing of composition of urea content due to the less formation of pores and lead to the densification occurred during the sintering process. These results is consistent with data obtained in previous research that stated that sintered density increase with the decreasing of urea content added up to the samples formulation [16].

In contrast, the porosity of the samples is decreased with increasing of SS316L powders into the formulations. The highest apparent porosity value at the sample with 40 wt.% of stainless steel powder was observed. The increasing amount of urea influence the apparent porosity of the samples [11]. The formation of larger pores due to the urea particles accumulated in one area and fully burnt off during the first stage of sintering process. This happens because the accumulated urea particles breaking the necks grow between particle contacts of stainless steel particles to form the larger size of pores thus increase the apparent porosity of the foams [11].

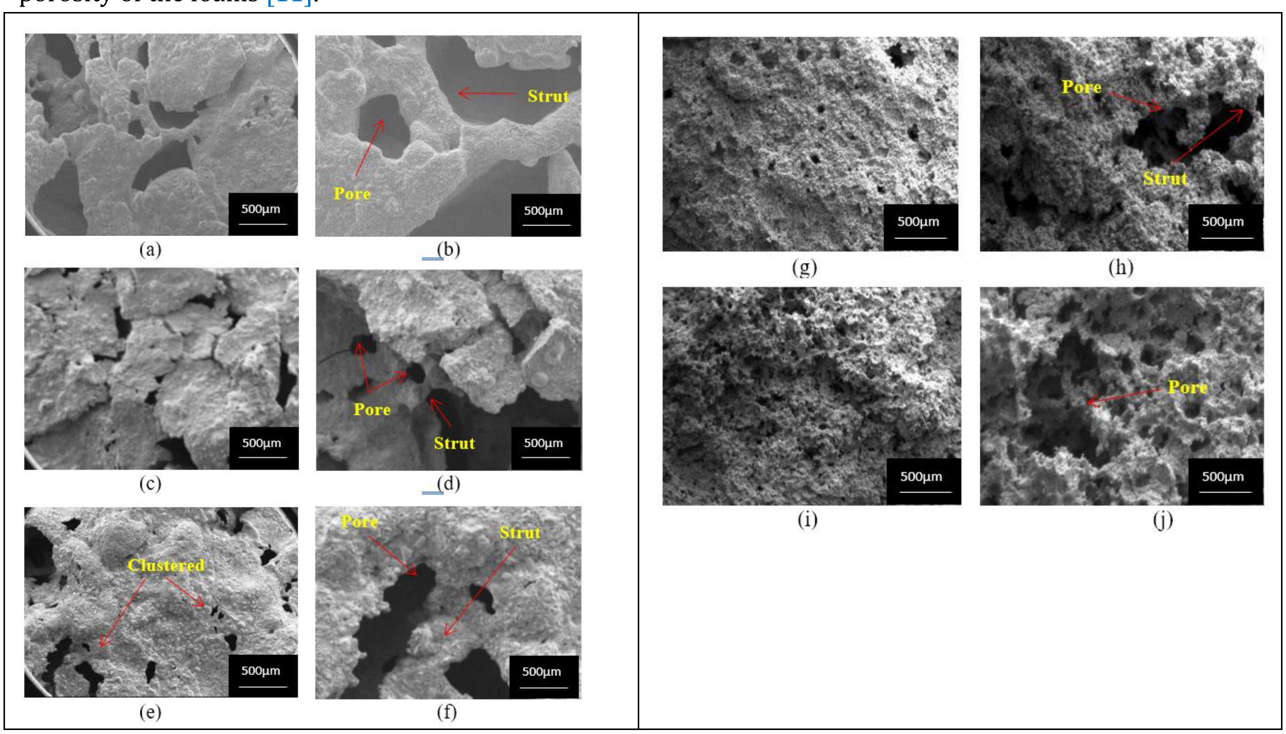

Fig. 1 Comparative SEM image for porous SS316L at composition of (a, b) 60 wt.%, (c, d) 55 wt.%, (e, f) 50 wt.%, (g, h) 45 wt.% and (i, j) 40 wt.% of urea content.

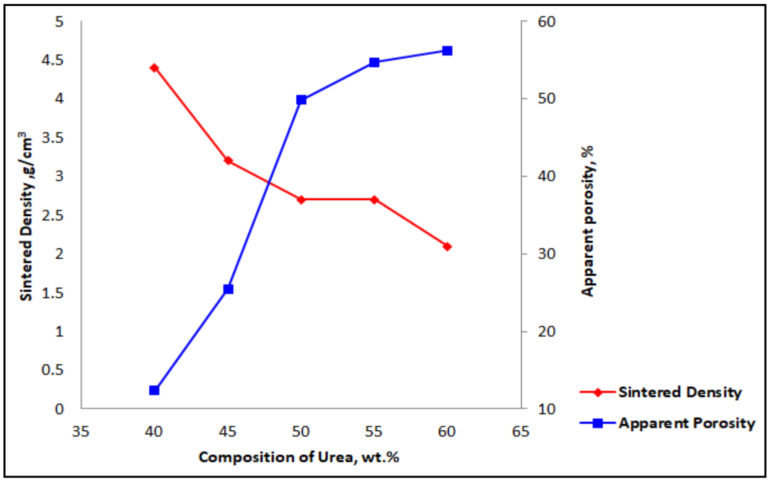

Fig. 2 Graph of sintered density and apparent porosity versus composition of urea

4. SUMMARY

Porous SS316L is successfully fabricated via space-holder technique and urea was used as a space holder material. Porous SS316L with 40 wt.% composition recorded the highest sintered density value of 4.36 g/cm^3 due to the decreasing of urea content into the formulation and allow densification occur between stainless steel particles. In contrast, porous SS316L with 60 wt.% of urea content produce highest apparent porosity of 56.22% compared to other compositions due to the highest amount of space holder into the formulation. It can be seen that the morphology of stainless steel foam with composition of 60 wt.% also forms larger pores with the struts clearly formed in the foams structures which is encouraging the cell in growth between the implant and the bones and thus have potential to be applied in biomedical application.

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