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Production of Stainless Steel Foams using the Powder Space Holder-Metal Injection Moulding (PSH-MIM) Method

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ABSTRACT

Background: Stainless steel foams have a great potential to be applied in various industries such as automotive, construction and biomedical. The primary method used to manufacture the stainless steel foams is powder metallurgy. **Objective:** The study attempts to produce the stainless steel foams using the Powder space holder-Metal injection moulding (PSH-MIM) method. The properties of the stainless steel foams were characterized and the effects of the volume fraction of the space holder on the stainless foams' properties were studies. **Results:** This study shows that the volume fraction of the space holder and the carbon content in the samples affect the properties of the stainless steel foams**.** The mechanical properties of the stainless steel foams are close to the mechanical properties of the human bones. **Conclusion:** The PSH-MIM method can be used to produce stainless steel foams for biomedical applications.

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INTRODUCTION

Metal foams are a new class material with superior physical and mechanical properties. Its unique properties include low specific weight, high gas permeability, and high thermal conductivity (Ashby *et al*., 2000). Recently, considerable attentions have been attracted on the stainless steel foams. Stainless steel is a metal alloy widely used in various applications such automotive industry, aerospace industry and construction. Due to its good corrosion resistance, stainless steel is used as biomedical implant. The pore structure forms in stainless steel foams alleviate the mismatch of elastic modulus between implants and the bone tissue. Consequently, the implant loosening is reduced, and therefore the implants shelf lives are extended. Moreover, stainless steels foam with an open – cellular pore aid in bone ingrowths and transportation of body fluid (Chen *et al*., 2009). Thus, stainless steel foams with adequate porosity and appropriate mechanical properties are deemed to be ideal bone implant.

Nowadays, there are many manufacturing methods can be used to produce metal foams. Traditionally, the simplest process to manufacture the metal foams is direct foaming of melts using gas. During foaming process, the inert gas is injected into a container of liquid metal. The foam structure form when molten metal solidified. Other foaming methods include investment casting or electroplating of metal vapour using polymers as precursor. Because these methods are based on molten metal or metal vapour, they are not suitable for high melting point metals such as stainless steels. Up to date, powder metallurgy (P/M) is still considered the most promising method to produce the stainless steel foams. The stainless steel foams are usually fabricated by loose metal powder sintering or powder space holder method (Mariotto *et al*., 2011; Bender *et al*., 2013). The newly developed PM method to fabricated stainless steel foams is Powder space holder- Metal injection moulding method (PSH-MIM). In PSH-MIM process, powder space holder which acts as a pore former is initially mixed with metal powder. The feedstock prepared is then used for the metal injection moulding process, involves the injection, debinding and sintering processes. With the proper selection and control of the space holder, the pore size and pore shape of the metal foams can be easily tailored. In addition, PSH-MIM process reduces the need of post-machining processes that could be detrimental to the integrity of the pore structure (Chen *et al*., 2009).

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Some studies have reported that the PSH-MIM successfully produced stainless steel foams (Nishiyabu *et al*., 2005; Gulsoy and German, 2008; Sunada *et al*., 2009 Manonukul *et al*., 2010). However, the stainless steel foams produced in these studies are not aim or not appropriate for the biomedical applications. To further explore the feasibility of PSH-MIM process on the production of stainless steel foams, this study will focus on production of stainless steel foams for biomedical applications. The physical and mechanical properties of stainless steel foams are characterized. The effect of the content of space holder on the stainless steel foams' properties were discussed and addressed.

MATERIALS AND METHODS

2.1 Materials:

316L stainless steel powder with a particle size of D_{90} = 45 μm and glycine powder with a particle size of D_{90} = 65 μm were used in this study. As shown in the Figure 1, both powders have irregular shape. Stainless steel powders with irregular shape particles provide strong interparticle frictions which retain the foam structure part shapes during the debinding and sintering process. Glycine was chosen as space holder due to its advantages of low cost and good chemical features. It is a non-toxic chemical and can be removed either by solvent extraction or thermal decomposition. In order to compare the effect of glycine on the properties of stainless steel foams, two different feedstocks with different volume fraction of glycine, 10% and 30% were prepared. A multi-component binder system consisting paraffin wax (60% wt) and polythene (40% wt) was selected for this study. The powders and binders were mixed with a sigma type blade mixer for 115 minutes at 140°C and rotor speed of 55 rpm. The feedstocks prepared were then granulated for the injection moulding process.

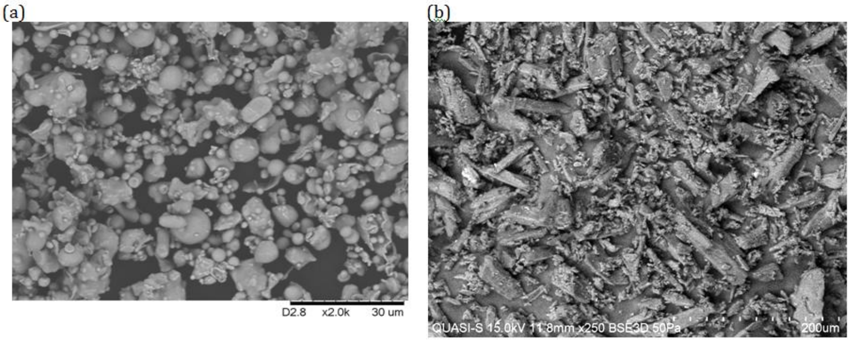

Fig. 1: Morphologies of initial powders: (a) Stainless steel powder (2000x); (b) Glycine(250X).

2.2 MIM process:

Injection moulding process was carried out by Battenfeld BA 250 CDC injection moulding machine. Cylindrical shape samples with a height of 20 mm and a diameter of 15 mm were prepared. The injection temperature was 130°C, mould temperature was 60°C, and injection pressure was 80 MPa. The paraffin wax and space holder of the specimen were removed by solvent debinding at 60°C for 36 h. Firstly; the green parts were immersed in heptanes to remove the paraffin wax. The space holder of the specimen was removed by distilled water. To remove the polythene and the residual space holder, the specimens were then heat treated at 250°C for 1 h and at 500°C for 1 h. Subsequently, the specimens were embed in a graphite powder bed, and sintered in a tube furnace with argon atmosphere at 1050°C, sintering time of 1 h. The graphite powder bed minimise the oxidation of the specimens.

2.3 Testing methods:

The microstructures of the sintered samples were characterized by scanning electron microscopy (SEM). Carbon content of the specimens was measured using CHNS analyzer. The density, general porosity and open porosity of the specimens were measured using Archimedes water immersion method, ISO 2738. Compression tests were carried out according to JIS H7902 standard using a 100-ton universal tensile testing machine with a strain rate of 2 mm/min. Five samples of each feedstocks were tested under a compression load.

RESULTS AND DISCUSSION

3.1 Microstructure characteristics and physical properties:

Figure 2 shows SEM images of sintered stainless steel foam samples on the surface. It can be seen that two types of pores i.e. micropores and macropores were observed in the sintered foam structure. The macropores have a pore size of approximately 50 μm. It is formed due to the removal of space holder. However, the small micropores are result from the incomplete sintering of the stainless steel powder. The interconnected macropores aid penetration of body fluid. In vivo, an interconnection size of more than 50 μm helps bone formation (Chen *et al*., 2009). As expected, the number of macropores in Sample 2 is greater than Sample 1 due to the larger content of space holder used.

The effect of fraction of space holder on the porosities and volumetric shrinkage is shown in Table 1. The general porosity of samples agrees with their microstructure characteristics. As amount of space holder increases, the general porosity increases. The open and closed porosities are also found to proportional to the space holder content. However, the results shown are inconsistent with the findings obtained by Mariotto *et al*. (2011), Mutlu and Oktay (2011), Bekoz and Oktay (2013). The main reason of this is due to the space holder used in this study has multimodal particles size distribution. As shown in Figure 1(b), the space holder used consists of a lot of tiny particle. These tiny particles are surrounded by stainless steel powder to form closed pores. When the volume fraction of space holder used increases, the amount of tiny particles increased. Thus, the closed porosity of Sample 2 is greater than Sample 1. The results in Table 1 also shows there is no significant different in volumetric shrinkage between the two samples. This is probably because of increased in closed porosity in Sample 2 hinder the sintering process; reduce the shrinkage.

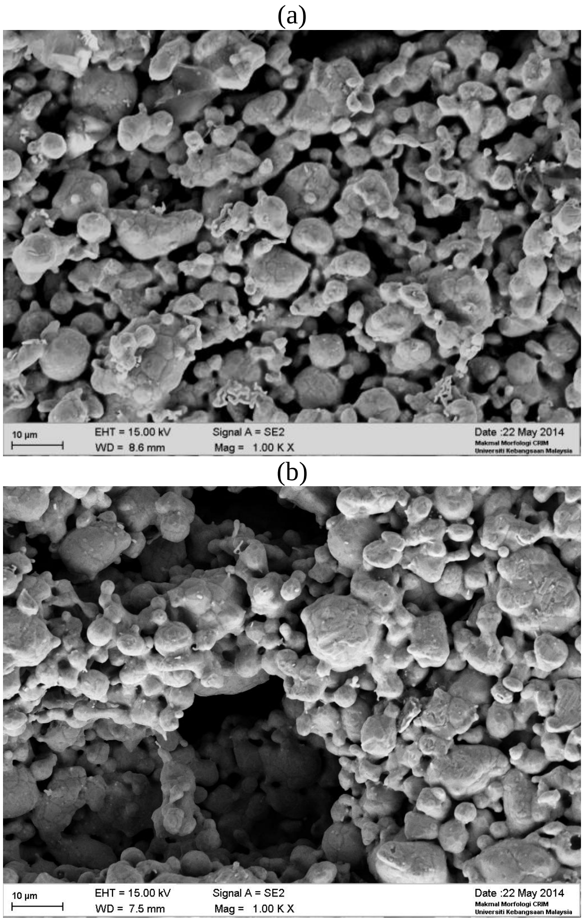

Fig. 2: SEM images of stainless steel foams for different samples: (a) Sample 1 (10% glycine); (b) Sample 2 (30% glycine).

Table 1: Porosities and volumetric shrinkage of the sintered stainless steel foams.

Stainless steel foams	Glycine Content (%)	General porosity (%)	Open porosity (%)	Closed porosity (%)	Volumetric shrinkage (%)
Sample 1	10	45.6 ± 3.05	34.1 ± 1.04	11.5 ± 0.67	10.0 ± 4.62
Sample 2	30	54.2 ± 4.76	33.4 ± 0.87	20.8 ± 1.04	11.7 ± 9.14

3.2 Mechanical properties:

The compressive stress-strain curves of stainless steel foams are shown in Figure 3. For typical metallic foam, compressive stress-strain curve is characterized by three regions: elastic region where stress increases linearly, a long plateau region with nearly constant flow stress, and a densification region with rapidly increasing flow stress (Surace *et al*., 2009; Mutlu & Oktay, 2011; Jha *et al*., 2013). According to Figure 3, the elastic regions of both samples occur from 0 to ~3% of normal strain. The curves bended which indicate begin of the plateau region. Nevertheless, the plateau region of both curves is not flat. It is probably due to the low porosities of the stainless foams. The higher porosity foam has a longer and flatter plateau region because the structure gives the chance to the cell walls for collapsing (Alizadeh & Mirzaei-Aliabadi, 2012). Furthermore, the plateau regions of the curves are serrated. This would be the consequence of the high carbon content (0.26%) in the samples. During compression, the samples walls tended to crack and fracture in a brittle manner. In general, the stainless steel foams produced exhibit the typical compressive behaviour of metallic foams.

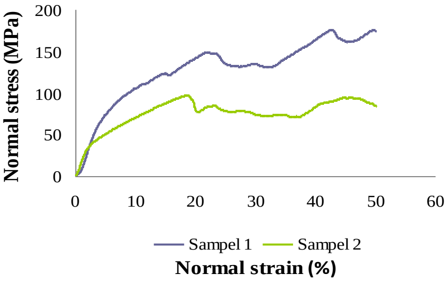

Fig. 3: Compressive stress-strain curves of stainless steel foams fabricated by MIM with different Glycine content in feedstocks.

Table 2: Mechanical properties of the sintered stainless steel foams.

Stainless steel foams	Compressive yield strength (MPa)	Elastic modulus (GPa)	Theoretical Compressive yield strength (MPa)	Theoretical Elastic modulus (GPa)
Sampel 1	53.9 ± 5.66	2.43 ± 0.787	20.7	57.1
Sampel 2	37.7 ± 3.79	2.05 ± 0.572	16.0	40.5

Table 3: Mechanical properties of cancellous bone.

Materials	Compressive yield strength (MPa)	Elastic modulus (GPa)
Femoral head	68	2.9
Femoral condyle	32	4.9
Vertebra	4.1	1.5

The mechanical properties of the samples are presented in Table 2. The theoretical value of mechanical properties can be derived via Gibson-Ashby model (Schüler *et al.*, 2013) Gibson and Ashby (1997) has expressed the relationships between the yield strength, elastic modulus and relative density for metal foams as follows:

$$\frac{\sigma_{pl}}{\sigma_{ys}} = (0.3)\left(\frac{\rho}{\rho_{ys}}\right)^{1.5} \qquad (1)$$

$$\frac{E_{pl}}{E_{ys}} = \left(\frac{\rho}{\rho_{ys}}\right)^{2} \qquad (2)$$

where ρ, σ_{pl} and E_{pl} are the density, yield strength and elastic modulus of the metal foam respectively, while the σ_{ys} and E_{ys} represents the corresponding mechanical properties. In this study, ρ_{ys} = 7.93 g/cm^3, σ_{pl}= 172 MPa and E_{ys} = 193 GPa, respectively (Dewidar *et al.*, 2007). The results in Table 2 indicate that the experimental values of compressive yield strength are greater than the theoretical value. This would be attributing from the high carbon content in the samples. The high carbon content also affected the elastic modulus by lowering its value. There are evidences that the high carbon and nitrogen content in stainless steel will lower the elastics modulus value (Salahinejad *et al.*, 2010; Dewidar, 2012). Sample 2 has higher porosity; therefore its compressive yield strength is lower than Sample 1. The similar finding also observed in elastics modulus. In designing the biomedical implants, the matching of elastic modulus between the implants and the bone tissue is the main concern. For comparison, the mechanical properties of cancellous bone referred from Xie *et al.* (2013) are listed in Table 3. It is noted that the mechanical propertied of the samples are close to those of the cancellous bone, especially the elastic modulus. Therefore, the negative effects caused by the mismatch of elastic modulus between implants and the bone tissue, could be avoided. In addition, the high compressive yield strength of the samples is presumably sufficient to support implantation and in vivo loading.

Conclusion:

Stainless steel foams with porosity ranging 37.7%- 53.9% were successfully manufactured via Powder space holder-Metal injection moulding (PSH-MIM) method. The samples produced display a compressive behaviour of metallic foams. The microstructure, physical properties and mechanical properties of the stainless foams are affected by the volume fraction of the space holder. High carbon content in the samples increases the yield strength but lowering the elastic modulus value. The stainless steel foams produced have great potential to be used in biomedical application as their mechanical properties are comparable with the mechanical properties of human cancellous bone.

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