Influence of Sintering Parameters on the Compressive Yield Strength of Stainless Steel Foams Produced by the Space Holder Method

(Pengaruh Parameter Pensinteran terhadap Kekuatan Alah Mampatan Keluli Tahan Karat Berbusa yang Dihasilkan melalui Kaedah Pengisi Pemegang Ruang)

TAN KOON TATT, NORHAMIDI MUHAMAD*, ANDANASTUTI MUCHTAR, ABU BAKAR SULONG & NEO MING CHERNG

ABSTRACT

Metallic foams are a new class of materials that have a great potential to be used in various functional and structural applications. Due to their competitive price compared to aluminium, metallic foams are anticipated to become an alternative material for light-weight structures. In this study, stainless steel foams are fabricated using a powder space holder method. The materials used include stainless steel powder, a novel space holder glycine and binders consisting of palm stearin and of polyethylene (PE). The stainless steel foams are sintered at 1100°C, 1200°C and 1300°C with sintering times of 1, 2 and 3 h, respectively, to investigate the effects of the sintering parameters on the compressive yield strength of the stainless steel foams. The results showed that all of the stainless steel foams produced exhibit the general behaviours of metal foams. The sintering time is the most significant parameter that influences the compressive yield strength of stainless steel foams. Increasing the sintering temperature and sintering time will increase the compressive yield strength. The interaction between the sintering temperature and sintering time is found to be not statistically significant.

Keywords: ANOVA; compressive strength; metal foams; space holder

ABSTRAK

Logam berbusa merupakan kelas bahan terbaru yang mempunyai potensi yang besar untuk digunakan dalam pelbagai aplikasi fungsi dan struktur. Disebabkan harganya yang kompetitif berbanding dengan aluminium, logam berbusa dijangka menjadi bahan alternatif untuk struktur berat-ringan. Dalam kajian ini, keluli tahan karat berbusa difabrikasi menerusi kaedah pemegang pengisi ruang. Bahan yang digunakan termasuklah serbuk keluli tahan karat, pemegang pengisi ruang terbaru, glisina dan bahan pengikat yang terdiri daripada stearin sawit dan polietilena (PE). Keluli tahan karat berbusa disinter pada suhu 1100°C, 1200°C dan 1300°C dengan masa pensinteran masing-masing 1, 2 dan 3 jam untuk mengkaji kesan parameter pensinteran terhadap kekuatan mampatan keluli tahan karat berbusa. Keputusan kajian menunjukkan bahawa semua keluli tahan karat berbusa yang dihasilkan menunjukkan perilaku umum logam berbusa. Masa pensinteran merupakan parameter yang paling signifikan mempengaruhi kekuatan mampatan. Interaksi antara suhu pensinteran didapati tidak ketara.

Kata kunci: ANOVA; busa logam; kekuatan mampatan; pemegang pengisi

INTRODUCTION

Metallic foams, a new class of materials, have recently gained considerable attention due to their excellent physical and mechanical properties. They are used in a wide range of applications, including lightweight structures; functional applications such as filtration, separation and heat or mass exchange; and sound or energy absorption. Many metals and metal alloys, such as aluminium, magnesium, titanium and their alloys, have been successfully foamed or made into cellular structures. Of these metals and metal alloys, aluminium is the most common and popular metal to have been foamed because of its ease of processing.

Recent developments in metallic foams have increased the need for developments in other materials. Stainless steel foam is one of the materials being developed. The physical and mechanical properties of stainless steel foam are comparable with those of aluminium foam. Its lower cost makes stainless steel foam a potential candidate for civil engineering and construction applications. Furthermore, stainless steel can also be used for medical implants because of its biocompatibility and high corrosion resistance. To date, the manufacturing of stainless steel foam is focused on powder metallurgy (PM) methods due to stainless steel's high melting temperature. One of the popular PM methods used to fabricate metal foams is the spacer holder method. In this method, metal powder is mixed with a space holder powder. After the parts have been shaped, the pores are formed by removing the space holder powder at low temperatures. Finally, the part is heat treated to obtain the desired properties. With the space holder method, the pore size and pore shape of the metal foams can be easily tailored.

There are a number of studies concerning the fabrication of metal foams via the space holder method (Alizadeh & Mirzaei-Aliabadi 2012; Esen & Bor 2007; Jiang et al. 2005; Jha et al. 2013; Kotan & Bor 2007; Surace et al. 2009). However, these studies are mainly focused on aluminium and titanium. Very little attention has been given to stainless steel. In order to fully understand the characteristics and properties of stainless steel foam fabricated via the space holder method, the present work examines the general compressive behaviour of stainless steel foam. The sintering parameters examined include the sintering temperature and sintering time. The pore size and pore shape distributions will be discussed in another publication.

EXPERIMENTAL DETAILS

Water-atomized 316 L stainless steel powder with a particle size of $D_{50} = 17 \ \mu m$ was used in this study. Glycine was used as the space holder. The space holder particles have an irregular shape and a size in the range of 220 to 617 μm . Glycine is a novel space holder and was chosen because of its low cost and its chemical properties. It is a non-toxic amino acid that can be produced with very high purity (Abdel et al. 2011). In addition, glycine is highly soluble in water and has a relatively low decomposition temperature. Therefore, it can be removed either by dissolution or thermal decomposition.

In order to increase the strength of the green compact, 9 wt. % of binders, consisting of palm stearin and polyethylene (PE), were added to the stainless steel and space holder mixture. The mixture was placed in a sigma type blade mixer for 105 min. Then, the homogenous mixture was uniaxially pressed at a pressure of 9 ton/m² into green compacts with sizes of 15 mm $\phi \times 20$ mm L. The green compacts were then immersed in heptane and distilled water for 8 h each to remove the binders and glycine. Prior to sintering, the green compacts were heat treated at 250°C for 2 h and at 500°C for 1 h to remove the residual binders and space holder. In order to study the influence of the sintering parameters, the green compacts were sintered at temperatures of 1100°C, 1200°C and 1300°C and with sintering times of 1, 2 and 3 h. Scanning electron microscopy was used to characterize the sintered samples. Finally, the relative densities of specimens were measured using Archimedes method and compression tests were carried out on the specimens at room temperature with a strain rate 2 mm/min.

RESULTS AND DISCUSSION

GENERAL BEHAVIOUR OF FOAMS

Stainless steel foams with porosities of 19-39% were fabricated. The scanning electron micrograph of the stainless steels foam is shown in Figure 1. Two types of pores are observed in the micrograph: Small isolated micropores and interpenetrated macropores. The macropores have a pore size of approximately 500 μ m and result from the removal of the space holder. The small micropores are formed due to incomplete sintering of the stainless steel powder (Bekoz & Oktay 2013). Interpenetrated macropores formed are very suitable for certain functional applications, such as filtration, separation and medical implants.

According to the literature (Dewidar et al. 2007; Esen & Bor 2007; Jha et al. 2013; Kotan & Bor 2007; Surace et al. 2009), the stress–strain behaviour of metallic foams is characterized by three distinct regions: Stress rising linearly with strain at low stresses (elastic deformation); a long plateau stage with small increases of flow stress yielding large strains; and a densification stage where the cell walls come into contact one with another, causing the flow stress to increase rapidly. Figure 2 shows the compressive deformation curves for the specimens with different porosities. The stainless steel foams produced



D2.7 x200 500 um

FIGURE 1. Scanning electron micrograph of stainless steels foam

exhibit typical compressive behaviour of metallic foams. However, the plateau stage of all the curves is not noticeable, which could be due to the low porosity of the stainless steel foam. The higher porosity foam structure would exhibit a longer and flatter plateau region because the structure gives the chance to the cell walls for collapsing and deforming (Alizadeh & Mirzaei-Aliabadi 2012).

Figure 2 also shows that the compressive yield strength of the stainless steel foams is inversely proportional to their porosity. The mechanical behaviour of metallic foams can be described using the Gibson-Ashby model (Ashby et al. 2000; Gibson & Ashby 1997; Schüler et al. 2013). Gibson and Ashby (Ashby et al. 2000; Gibson & Ashby 1997) showed the relationship of relative stress and relative density for cellular materials as follows:

$$\frac{\sigma_{pl}}{\sigma_{ys}} = C \left(\frac{\rho}{\rho_{ys}}\right)^{1.5},\tag{1}$$

where ρ is the density of the foam; ρ_{y_s} is the density of the bulk material; σ_{pl} is the yield stress of the foam; σ_{y_s} is the yield stress of the bulk material; and C is a constant of 0.3 for cellular metals and polymers.

The nominal yield strength of the fully dense 316 L stainless steel is 172 MPa (Dewidar et al. 2007). Figure 3 shows the effect of porosity on the compressive yield strength of the stainless steel foams fabricated in this study. Both experimental and theoretical data show decreases in the compressive yield stress with increasing porosity. Furthermore, all theoretical stress data are higher than the experimental data. This result is a consequence of different deformation behaviour between the theoretical hypothesis and the experimental (Dewidar et al. 2007; Wen et al. 2001). However, the deviation between theoretical stress and experimental stress becomes smaller as the porosity increases. Hence, the Gibson-Ashby model best describes the compressive behaviour of the stainless steel foams when the porosity exceeds 30%.

EFFECT OF SINTERING PARAMETERS ON COMPRESSIVE YIELD STRENGTH

In order to study the effect of the sintering parameters, nine experiments (five replications of each experiment) with different sintering temperatures and sintering times were performed. The analysis of variance (ANOVA) technique was used to draw meaningful conclusions from the data. From the ANOVA results shown in Table 1, the relative significance and the effects of the sintering parameters were



FIGURE 2. Compressive stress-strain curves for stainless steel foams with different porosities (sample with porosity 19% carried out at 2 h, 1200°C; porosity 22% carried out at 3 h, 1100°C and porosity 39% carried out at 1 h, 1100°C)



FIGURE 3. Effect of porosity on the compressive yield strength of stainless steel foams

TABLE 1. ANOVA	table fo	r compressive	yield strength
----------------	----------	---------------	----------------

-					
Source	DF	SS	MS	F	<i>p</i> -value
Sintering Temperature	2	89.681	44.841	2.41	0.104
Sintering Time	2	133.648	66.824	3.60	0.038
Interaction	4	19.083	4.771	0.26	0.904
Error	36	668.628	18.573		
Total	44	911.040			

*DF, degrees of freedom; SS, sum of squares; MS, mean square

determined. Both sintering temperature and sintering time have a significant effect on the compressive yield strength at a 90% significance level. The most significant factor is shown by the much lower p-value.

The response graphs presented in Figure 4 shows that a higher sintering temperature yields higher compressive yield strength. At a sintering temperature of 1100°C, the stainless steel foam displays low compressive yield strength. The main reason for this result is an insufficient sintering process at low sintering temperatures. Insufficiently sintered PM materials have low mechanical properties (Dewidar 2012; German 1996). The SEM image shown in Figure 5 clearly indicates that the number of pores in stainless steel foam is greater with sintering at 1100°C compared with sintering at 1300°C. Although a sintering temperature of 1100°C has promoted neck formation, the bond formed is weak and the powder particles cannot fully bond to each other. The bonds formed among powder grain boundary are weak if the powder particles cannot form exact bond for each other (Dewidar 2012). When temperature increases to 1300°C, the grains diffuse quickly along the boundary, causing the elimination of the pores and densification of the part. Thus, increasing the sintering temperature will increase the compressive yield strength of stainless steel foam.

Yoon et al. (2003) showed that increasing the sintering time and temperature does not change the yield strength of 316 L stainless steels because of grain coarsening. Conversely, in this study, the stainless steel foam exhibited increased compressive yield strength with increasing sintering time. The result of this study is consistent with the results obtained by Mutlu and Oktay (2013) and Salahinejad et al. (2010). As discussed previously, the compressive yield strength of stainless steel foam is affected by its relative density. German (1996), in his model describing the sintered fractional density, has noted that increasing the sintering time will increase the density of the part. An increased sintering time results in the parts having a longer exposure time to sintering activity, which results in densification of the part and a decrease in the number of pores. Consequently, increasing the sintering time will improve the mechanical behaviour of stainless steel foam. Despite the increase in compressive yield strength with increased sintering temperature and time, the stainless steel foam sintered at 1300°C for 3 h exhibited a lower strength compared with the stainless steel foam sintered at 1200°C for 3 h. When the part is sintered at 1300°C for 3 h, small pores coalesce, leading to the growth of larger pores, which retards the sintering activity. The growth of large pores in a metal foam structure will decrease the compressive yield strength of the metal foam (Ahmad et al. 2010). The SEM image in Figure 6 shows pore coalescence occurring when the part is sintered at 1300°C for 3 h. Although this



FIGURE 4. Main effect plot for the compressive yield strength of stainless steel foams



FIGURE 5. SEM images of stainless steel foams sintered at different temperature for 1 h (a) 1100°C and (b) 1300°C



D3.7 x1.0k 100 um

FIGURE 6. SEM image of stainless steel foam sintered at 1300°C for 3 h

observation suggests that there is an interaction between the sintering temperature and sintering time, the ANOVA results indicate that this interaction is not statistically significant. In general, increasing the sintering temperature and sintering time will increase the compressive yield strength of stainless steel foams.

CONCLUSION

Stainless steel foams with porosities in the range of 19-39% were successfully manufactured via the powder space holder method. All stainless steel foams show the compressive behaviour of metallic foams. From this study, it can be concluded that the Gibson-Ashby model is suitable for describing the mechanical properties of stainless steel foams when the porosity is greater than 30%. There is significant evidence that the sintering temperature and sintering time affect the compressive yield strength of stainless steel foams. As the sintering temperature and sintering time increase, the compressive yield strength will increase. However, sintering at 1300°C for 3 h will cause pore coalescence. Finally, the interaction between sintering temperature and sintering time was found to not be significant to compressive yield strength. By using the Gibson-Ashby model and

controlling the sintering conditions, stainless steel foams with various strengths can be fabricated to fit the required functional applications.

ACKNOWLEDGEMENTS

This work is supported by Universiti Kebangsaan Malaysia research grant, UKM-AP-NBT-11-2009.

REFERENCES

- Abdel, G.N.A., El-Shenawy, A.E. & Hussien, W.A.M. 2011. The inhibitive effect of some amino acids on the corrosion behaviour of 316 L stainless steel in sulfuric acid solution. *Modern Applied Science* 5(4): 19-29.
- Ahmad, S., Muhamad, N., Muchtar, A., Sahari, J., Jamaludin, K.R., Ibrahim, M.H.I., Mohamad Nor, N.H. & Murtadhahadi. 2010. Pencirian titanium berbusa yang dihasilkan pada suhu pensinteran yang berbeza menggunakan kaedah buburan. *Sains Malaysiana* 39(1): 77-82.
- Alizadeh, M. & Mirzaei-Aliabadi, M. 2012. Compressive properties and energy absorption behavior of Al-Al₂O₃ composite foam synthesized by space-holder technique. *Materials and Design* 35: 419-424.
- Ashby, M.F., Evans, A., Fleck, N.A., Gibson, L.J., Hutchison, J.W. & Wadley, H.N.G. 2000. *Metal Foams-Design Guide*. US: Butterworth- Heinemann.

- Bekoz, N. & Oktay, E. 2013. Effect of heat treatment on mechanical properties of low alloy steel foams. *Materials* and Design 51: 212-218.
- Dewidar, M. 2012. Influence of processing parameters and sintering atmosphere on the mechanical properties and microstructure of porous 316 L stainless steel for possible hard-tissue applications. *International Journal of Mechanical* & Mechatronics Engineering 12(1): 10-24.
- Dewidar, M., Khalil, K.A. & Lim, J.K. 2007. Processing and mechanical properties of porous 316 L stainless steel for biomedical applications. *Trans. Nonferrous Met. Soc. China* 17: 468-473.
- Esen, Z. & Bor, S. 2007. Processing of titanium foams using magnesium spacer particles. *Scripta Materialia* 56: 341-344.
- German, R.M. 1996. *Sintering Theory and Practice*. New York: Wiley.
- Gibson, L.J. & Ashby, M.F. 1997. *Cellular Solids: Structure and Properties [M]*. Cambridge: Cambridge University Press.
- Jha, N., Mondal, D.P., Majumdar, J.D., Badkul, A., Jha, A.K. & Khare, A.K. 2013. Highly porous open cell Ti-foam using NaCl as temporary space holder through powder metallurgy route. *Materials and Design* 47: 810-819.
- Jiang, B., Zhao, N.Q., Shi, C.S., Du, X.W., Li, J.J. & Man, H.C. 2005. A novel method for making open cell aluminium foams by powder sintering process. *Materials Letters* 59: 3333-3336.
- Kotan, G. & Bor, A.S. 2007. Production and characterization of high porosity Ti-6Al-4V foam by space holder technique in powder metallurgy. *Turkish J. Eng. Env. Sci.* 31: 149-156.
- Mutlu, I. & Oktay, E. 2013. Mechanical properties of sinterhardened Cr–Si–Ni–Mo based steel foam. *Materials and Design* 44: 274-282.
- Salahinejad, E., Amini, R., Marasi, M. & Jafar, H.M. 2010. The effect of sintering time on the densification and mechanical properties of a mechanically alloyed Cr–Mn–N stainless steel. *Materials and Design* 31: 527-532.

- Schüler, P., Fischer, S.F., Bührig-Polaczek, A. & Fleck. C. 2013. Deformation and failure behaviour of open cell Al foams under quasistatic and impact loading. *Materials Science & Engineering A* 587: 250-261.
- Surace, R., De Filippis, L.A.C., Ludovico, A.D. & Boghetich, G. 2009. Influence of processing parameters on aluminium foam produced by space holder technique. *Materials and Design* 30: 1878-1885.
- Wen, C.E., Mabuchi, M., Yamada, Y., Shimojima, K., Chino, Y. & Asahina. 2001. Processing of biocompatible porous Ti and Mg. Scripta Materialia 45: 1147-1153.
- Yoon, T.S., Lee, Y.H., Ahn, S.H., Lee, J.H. & Lee, C.S. 2003. Effects of sintering conditions on the mechanical properties of metal injection molded 316 L stainless steel. *ISIJ Int.* 43: 119-126.

Tan Koon Tatt, Norhamidi Muhamad, Andanastuti Muchtar, Abu Bakar Sulong & Neo Ming Cherng Department of Mechanical and Material Engineering Faculty of Engineering and Built Environment Universiti Kebangsaan Malaysia 43600 UKM Bangi, Selangor Darul Ehsan Malaysia

Tan Koon Tatt Faculty of Pharmacy MAHSA University Pusat Bandar Damansara, Damansara Heights 50490 Kuala Lumpur Malaysia

*Corresponding author; email: norhamidi@ukm.edu.my

Received: 19 March 2015 Accepted: 3 November 2015