

## Rheological Behaviour of Novel Feedstock for Manufacturing Porous Stainless Steel via (MIM)-PSH

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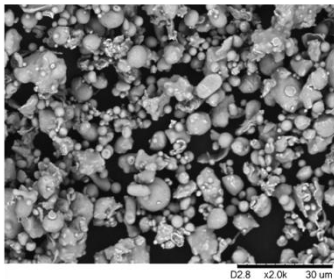
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### Graphical abstract



### Abstract

Metal foam has emerged as a new class material that can be used in structural and functional applications. Because of its excellent mechanical and physical properties, it has been extensively used in aerospace, automotive and medical industries. There are several ways to produce the metal foams. In this study, a net shape foaming technology namely Metal injection moulding-Powder space holder method (*MIM-PSH*) was used to produce the porous metal. A novel space holder, glycine was mixed with the water atomized stainless powder, palm stearin and polyethylene binder. Rheological behaviours of the feedstocks were fully investigated. The volume fraction of glycine was varied for 50% and 70%, to study its effect on the rheological properties. The results showed that all feedstocks exhibit shear thinning behaviour. As the volume fraction of space holder increased, the viscosities of feedstocks are increased. The activation energy,  $E$  is proportional to the amount of space holder used. All feedstocks are found to be suitable for MIM-PSH to produce the porous stainless steel.

**Keywords:** Metal injection moulding; metal foams; space holder; rheological properties

### Abstrak

Logam berbusa telah berkembang sebagai suatu kelas bahan terbaru yang boleh digunakan dalam aplikasi struktur dan fungsi. Disebabkan sifat-sifat mekanikal dan fizikalnya adalah sangat baik, ia telah digunakan secara meluas dalam industri aeroangkasa, otomotif dan perubatan. Terdapat pelbagai cara untuk menghasilkan logam berbusa. Dalam kajian ini, teknologi pembusaan yang bernama Pengacuanan suntikan logam-Bahan pengisi pemegang ruang (*MIM-PSH*) telah digunakan untuk menghasilkan logam berbusa. Bahan pengisi pemegang ruang yang terbaru, *glycine* telah dicampurkan dengan serbuk keluli tahan karat berpengatoman air, pengikat stearin sawit dan polietena. Sifat-sifat reologi untuk bahan suapan ini telah dikaji sepenuhnya. Pecahan isipadu *glycine* adalah diubahkan 50% dan 70% untuk mengkaji kesannya terhadap sifat-sifat reologi. Keputusan kajian menunjukkan bahawa semua bahan suapan mempamerkan sifat penipisan ricih. Peningkatan kandungan isipadu bahan pengisi pemegang ruang meningkatkan kelikatan bahan suapan. Tenaga pengaktifan,  $E$  adalah berkadaran dengan jumlah bahan pengisi pemegang ruang yang digunakan. Semua bahan suapan didapati sesuai untuk MIM-PSH bagi menghasilkan keluli tahan karat berporos.

**Kata kunci:** Logam berbusa; pemegang ruang; pengacuanan suntikan logam; sifat reologi

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### 1.0 INTRODUCTION

Metallic foams and porous metals are new class of material that gains a considerable attention due to their excellent and superior properties. Metal are tough, strong and has good thermal and electrical conductivity. These features make the metal foams are become more popular than polymeric foam. Typical usage of metal foams and porous metals include filters for separating solids from liquids and gases, electrode in chemical and fuel cell and for

sound dampening and attenuation. The interconnected pores also allow the porous metals can be used as biomedical implant.

As stated by Banhart, the fabrication methods of metal foam can be classified into four groups: liquid, solid, gas and aqueous solutions [1]. From economic point of view, the liquid state processing route will be selected when metal's melting point is low. For metals having high melting point, such as titanium and stainless steel, solid state processing through powder metallurgy (P/M) would be the most promising foaming method to date. Among the solid state processes used to fabricate metal foams are

loose powder sintering, hollow powder sintering, sintering of powders with a fugitive scaffold and powder sintering with entrapped gas. Indeed, these processes can produce metal foam with the desired mechanical properties. However, these methods have shortcomings in terms of difficulty to control porosity. In addition, the pores size and pore shape is also difficult to control because it is entirely depend on the size and shape of host powder used. Precise control of porosity as well as size and pore structure is very crucial in filtration systems industry and medical implants. Other newly developed solid state processes like rapid prototyping, laser sintering are deemed to be good processes in controlling the porosity, but have weakness in dimensional control of products.

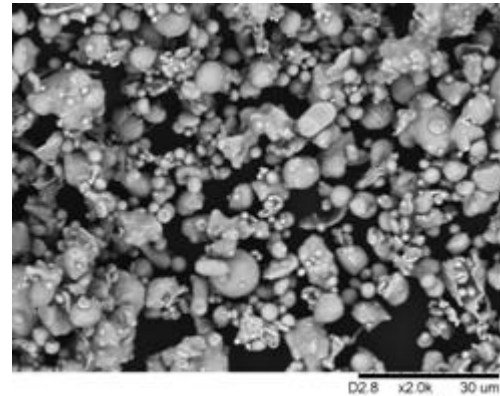
One of the most popular solid state processes which capable to control the porosity of metal foams is powder space holder (PSH) method. In the powder space holder method, metal powder is mixed with a space holder that normally decomposes at low temperatures. Space holder acts as an intermediary which holds the space between the metal powders. Once the space holder removed, the pores can be produced. Space holder that commonly used includes carmabide, ammonium bicarbonate, and sodium chloride. Generally, the PSH can be used in both conventional P/M and MIM. MIM is used when the product design is complex. Additionally, MIM also offers an opportunity to produce mass production.

There are number of studies on PSH with various material has been published, for example, aluminium [2], titanium superalloy [3], and copper [4]. Nevertheless, most of the studies were focused on the P/M techniques. MIM-PSH literature is limited because it is a relatively new process to fabricate the metal foams and porous metal. Production of porous metal parts on a commercial basis using MIM-PSH studied by Nishiyabu et al. [5]. They used the average size of 10  $\mu\text{m}$  and 40  $\mu\text{m}$  PMMA as space holder to produce micro-parts porous 316L. Manonukul et al. using the same type of space holder to produce a close-cell stainless foam [6]. In another study, porous titanium implants were successfully injection moulded using sodium chloride as space holder [7]. All previous studies on MIM-PSH have only focus on the effect of space holder on the mechanical properties of sintered part. Rheological properties of MIM-PSH feedstocks have not been extensively reported. Therefore, the objective of this study is to investigate the effect of space holder on the rheological properties of the MIM- PSH feedstocks. The influences of different volume fraction of space holder was addressed and discussed.

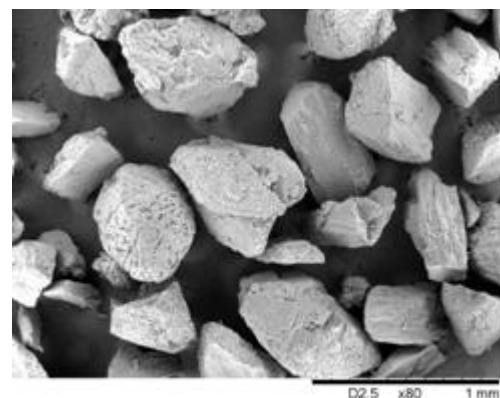
## 2.0 EXPERIMENTAL PROCEDURE

In this work, 316L stainless steel powder with particle size of  $D_{50} = 17 \mu\text{m}$  were used. As shown in Figure 1, 316L powders are ligament in shape. Ligament shapes have imparted strong

interparticle friction which retains the parts' shape during the leaching and removal of the space holder. Glycine was chosen as space holder because of its advantage of low cost and its chemical features. It is non-toxic, relatively cheap and easy to produce with very high purities [8]. Table 1 clearly shows that glycine has a higher melting temperature and decomposition temperatures than binders. This indicates glycine will not melt, vaporize or decompose during the moulding process. The glycine particles were sieved to the size range 220  $\mu\text{m}$  to 617  $\mu\text{m}$ . Binder used consisted of 70% wt of palm stearin and 30% wt of polyethylene (PE). In order to study the effect of space holder to rheological behaviours, the binder solid loading was kept constant at 40% vol while the volume fraction of glycine was varied.



(a)



(b)

Figure 1 SEM image (a) SS 316L (2000x), (b) Glycine (80x)

Table 1 Materials properties and fraction of compound

Material	Particle Size ( $D_{50}$ ), $\mu\text{m}$	Density, $\text{gcm}^{-3}$	Melting temperature, $^{\circ}\text{C}$	Decomposition temperature, $^{\circ}\text{C}$	Volume fraction	
					MIM feedstock	Porous compound
SS 316L	17	6.44	1375	---	60 vol.%	
Palm stearin	---	0.89	61	288	40 vol.%	30 vol.% & 50 vol.%
Polyethylene	---	0.95	127	390		
Glycine	376	1.39	236	236	---	50 vol.% & 70 vol.%

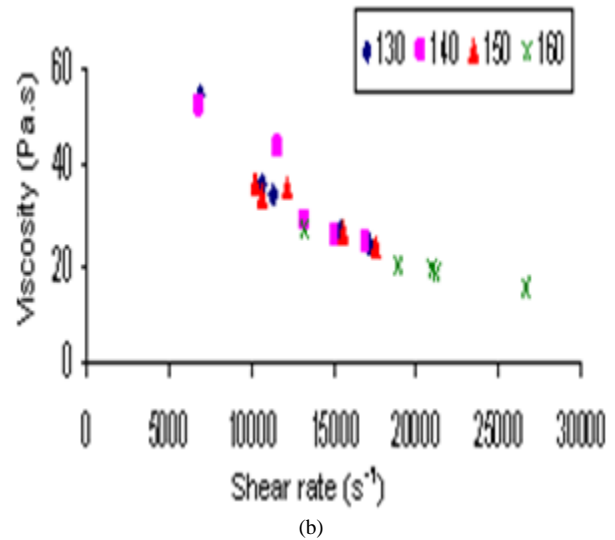
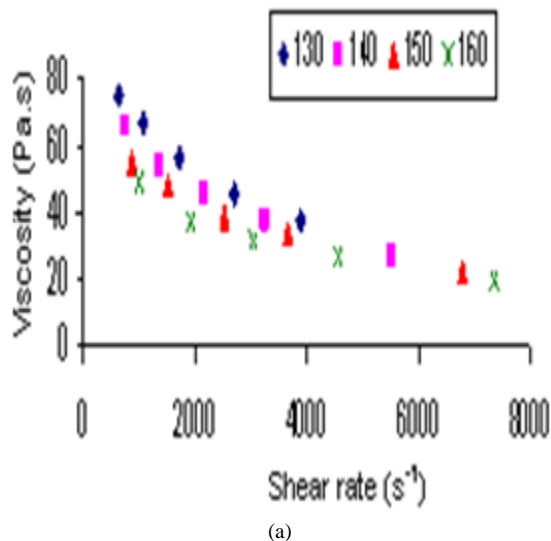
First, the glycine was mixed with 316L powder in sigma type blade mixer for 15 minutes. The powder particles adhered to the surface of the space holder. The binder were then added and fully mixed for 90 minutes at 140°C and rotor speed of 55 rpm. Mixture was granulated with a strong crusher. Two compositions of feedstocks with defined porosity of 50% volume fraction (V50) and 70% volume fraction (V70) were prepared. After mixing and granulation process, the feedstocks viscosity and rheological characteristics was measured using a Shimadzu 500-D capillary rheometer. The samples were extruded through a die with L/D of 10. The test was carried out with various capillary temperatures ranging from 130°C to 160°C and test load between 3kgf to 16.5kgf.

### 3.0 RESULTS AND DISCUSSION

In MIM process, the viscosity change during moulding is the main factor determining the success of the process. Rheological behaviours of the feedstock provide a good overview about its flow behaviours during the moulding process. Theoretically and practically reported by previous researches, the suitable range of viscosity for MIM feedstock is between 10 Pa.s to 1000 Pa.s and of shear rate range from 100 s<sup>-1</sup> to 1000 s<sup>-1</sup> [9],[10]. It is important to study the trend of the viscosity in different shear rate because viscosity is the most important indicator of feedstocks quality that influences the successful of moulding stage [10]. The flow behaviours of the fluid can be evaluated using the flow behaviours index,  $n$ . For non-Newtonian fluid, relationship between viscosity and shear rate can be expressed by following equation:

$$\eta = K \dot{\gamma}^{n-1} \quad (1)$$

where  $\eta$  is the viscosity,  $K$  is the constant and  $n$  is the flow behaviours index. The  $n$  value is used to evaluate the dependency of viscosity to shear rate. Non-Newtonian fluid viscosity varies depending on shear rate changes. Feedstock shows pseudo-plastic behaviour ( $n < 1$ ), the viscosity decrease with increased shear rate is desired in MIM process.



**Figure 2** Correlation of viscosity and shear rate at different content of space holder (a) V50 (50% vol), (b) V70 (70% vol)

**Table 2** Comparison of flow behavior index at different content of space holder

Feedstock	Temp(°C)			
	130	140	150	160
V50	0.6056	0.5626	0.5539	0.5616
V70	0.1158	0.1029	0.1885	0.1755

Figure 2(a) and 2(b) show the correlation of viscosity and shear rate at different volume fraction of glycine. All feedstocks exhibit a pseudo-plastic behaviour in which viscosity is decreased with increasing shear rate. Feedstock V50 can be extruded using low temperature and low pressure compared to feedstocks V70. For V70, a very high shear rates are required to give a similar range of viscosity as V50. This indicates the viscosities of feedstocks were increased with increased content of space holder. When binders melted, the metal powder with smaller particle size filled the void between the space holders. Consequently, more binders are released and therefore increased the flowability of the feedstock. Feedstock V70 with greater content of space holder required more binders to fill the void between the space holders. Therefore, feedstock V70 is more viscous than V50 at the same temperature range. In addition, as shown in Figure 2(b) the viscosity change inconsistent with the shear rate at 150 °C and 160 °C. This is possibly due to powder-binder separation had occurred at high temperature.

Table 2 shows the comparison of flow behaviour index at different content of space holder. At first, the flow behaviour index,  $n$  for both feedstocks decreases with the increase of the temperature. Ibrahim et al. (2009) demonstrated the similar finding for the SS316L feedstocks. Nevertheless, flow behaviour index is found to be increased when temperature is being increased to 150 °C and 160 °C [4]. This observation is in line with the inconsistently of the viscosity at high temperature. Furthermore, Table 2 clearly displays the effect of space holder

content on the flow behaviour index. The flow behaviour index,  $n$  is decreased with the increased of space holder content. This is due to a small shear stress is required to overcome a weak interparticle force between glycine particles. Accordingly, the viscosity change is more noticeable when space holder content is increased. Despite of high viscosities value, feedstock with 70% space holder, V70 shows more sensitivity to shear or greater pseudo-plastic behaviour compared to feedstock with 50% space holder V50. Although high shear sensitivity is important to produce a complex and delicate parts, a very low value of  $n$  may cause defects such as binder separation and jetting occur during moulding process. Thus, the feedstock V50 is a better feedstock for injection moulding in term of consideration of flow behaviour index.

Temperature dependency of viscosity is another essential factor that influences the flow behaviour of feedstock. In order to evaluate the effect of temperature, the flow activation energy,  $E$  was computed using Arrhenius equation as shown:

$$\eta = \eta_o \exp\left(\frac{E}{RT}\right). \quad (2)$$

where  $R$  is the gas constant,  $T$  is the temperature in Kelvin unit,  $\eta$  is the mixture viscosity and  $\eta_o$  is the viscosity at reference temperature.

In MIM, small value of  $E$  or less sensitivity of viscosity to temperature is preferred. Higher value of  $E$  may cause viscosity change rapidly with changed temperature. During the moulding stage, the sudden change of viscosity will induce internal stresses in part and lead to crack occurs. As can be seen in Table 3, all feedstocks have very low activation energy. The  $E$  values obtained in this work are lower than  $E$  values obtained in previous works [11–13]. This results from the use of larger particle size in this experiment. In fact, small particles have a larger surface energy that allows heat to dissipate more quickly to flow. Besides that, the viscosity observed more sensitive to temperature at high shear rate. A comparison of  $E$  value of both feedstocks shows that the viscosity of feedstock with 70% glycine, V70 is more sensitive to temperature. This is by reason of there are more space holder disperse in powder-binder matrix in V70 which causes heat can be dissipated to more extend in the flow. However, there is an evidence indicate binder separation had occur in V70 at high shear rate as  $E$  value is decreased with increasing shear rate.

**Table 3** Comparison of activation energy ( $E$ ) and moldability index ( $\alpha_{stv}$ ) (shear rate= 1000  $s^{-1}$ ) at different content of space holder

Feedstock	E(kJ/mol)		$\alpha_{stv}$
	shear rate =100 $s^{-1}$	shear rate =1000 $s^{-1}$	
V50	8.61	13.39	4538
V70	24.21	15.37	1700

Beside flow behaviour index and  $E$ , the moldability index,  $\alpha_{stv}$  was computed to access the general flow behaviour of feedstock. The higher the value of  $\alpha_{stv}$ , the feedstock will

possess better rheological properties. In general, the equation used to describe the moldability index is the simplified equation from Weir's model [14].

$$\alpha_{stv} = \frac{1}{\eta_o} \frac{|n-1|}{E/R}. \quad (3)$$

Table 3 indicate the moldability index is proportional to the space holder content. In the absence of defect such as jetting or binder separation, the higher value of  $\alpha_{stv}$  is desirable. It clearly indicates that feedstocks V50 give the highest moldability index. Therefore it is a better feedstock use for MIM-PSH. Likewise, feedstock V50 has a better powder-binder ratio to get quick powder repacking and binder molecule orientation during moulding process [11], [15].

#### 4.0 CONCLUSION

The rheological behaviour of novel feedstock for porous 316L stainless steel has been investigated. All feedstocks are possible to be injection moulded as flow behaviour index indicates the shear thinning behaviour. The flow behaviour index, activation energy and moldability index are proportional to the content of space holder used. The sensitivity of feedstock's viscosity to the shear rate and temperature is increase with increased of content of space holder. Feedstock V70 posses the high viscosity as more void was formed with greater amount of space holder. Hence, the viscosity of feedstock is increases with increasing space holder content. More binder should be added in higher volume fraction of space holder. Further investigations are required on mixing steps in order to increase the flowability of feedstock with high volume fraction of space holder. Feedstock V50 is a better candidate for MIM-PSH in term of the flow behaviour.

#### Acknowledgement

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#### References

- [1] Banhart, J. 2000. Manufacturing Route for Metallic Foams. *Journal of the Mineral Metal & Material Society*. 52(12): 22–27.
- [2] 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.
- [3] Esen, Z. & Bor, S. 2007. Processing of Titanium Foams Using Magnesium Spacer Particles. *Scripta Materialia*. 56: 341–344.
- [4] Zhang, E., Wang, B. 2005. On the Compressive Behaviour of Sintered Porous Coppers with Low to Medium Porosities. Part I. Experimental Study. *International Journal of Mechanical Sciences*. 47: 744–756.
- [5] Nishiyabu, K., Matsuzaki, S., Tanaka, S. 2007. Net-shape Manufacturing of Micro Porous Metal Components by Powder Injection Molding. *Materials Science Forum*. 534–536: 981–984.
- [6] Manonukul, A., Muenya, N., Léaux, F. & Amaranan, S. 2010. Effects of Replacing Metal Powder with Powder Space Holder on Metal Foam Produced by Metal Injection Moulding. *Journal of Materials Processing Technology*. 210: 529–535.
- [7] Chen, Liang-jian., Li, Ting., Li, Yi-min., He, Hao & Hu, You-hua. 2009. Porous Titanium Implants Fabricated by Metal Injection Molding. *Trans. Nonferrous Met. Soc. China*. 19: 1174–1179.
- [8] Abdel Ghanyl, N. A., El-Shenawy, A. E. & Hussien, W. A. M. 2011. The inhibitive effect of some amino acids on the corrosion behaviour of 316L stainless steel in sulfuric acid solution. *Modern Applied Science* 5, 4: 19-29.

- [9] German, R. M. & Bose. 1997. *Injection Moulding of Metal and Ceramics*. Princeton. New Jersey: Metal Powder Industries Federation.
- [10] Khakbiz, M., Simchi, A. & Bagheri, R. 2005. Analysis of the rheological behavior and stability of 316L stainless steel-3 wt% TiC powder injection molding feedstock. *Materials Science and Engineering A*. 407: 105–113.
- [11] Ibrahim, M. H. I., Muhamad, N. & Sulong, A. B. 2009. Rheological investigation of water atomized stainless steel powder for micro metal injection moulding. *International Journal of Mechanical and Materials Engineering (IJMME)*. 4(1): 1–8.
- [12] Yimin, Li., Liu Jun Li & Khalil, K. A. 2007. Effect of powder loading on metal injection molding stainless steels. *Journal of Materials Processing Technology*. 183: 432–439
- [13] Jamaludin, K. R., Muhamad, N., Amin, S. Y. M., Rahman, M. N. Ab. & Murtadhahadi. 2008. Rheological behavior of SS316L gas atomized powder in bimodal particle size distribution in a composite binder system. *International Journal of Mechanical and Materials Engineering (IJMME)*. 3(2): 108–114.
- [14] Weir, F. E., Doyle, M. E & Norton, D. G. 1963. Mouldability of plastics based on melt rheology. *SPE Transactions* 3: 32–336.
- [15] Hooman Abolhasani. & Noorhamidi Muhamad. 2009. Rheological investigation of a starch-based binder and feedstock for metal injection moulding. *International Journal of Mechanical and Materials Engineering (IJMME)*. 4(3): 294–299.