Characterization of Stainless Steel 316L Feedstock for Metal Injection Molding (MIM) Using Waste Polystyrene and Palm Kernel Oil Binder System

R. Asmawi 1, M.H.I Ibrahim 1, A. M. Amin 1, N. Mustafa 1

1 Advanced Manufacturing and Materials Center (AMMC), Universiti Tun Hussein Onn Malaysia (UTHM), 86400 Parit Raja, Batu Pahat, Johor, Malaysia

E-mail: roslias@uthm.edu.my

Abstract. This paper presents the homogeneity characterisation of MIM feedstock consisting Stainless steel alloy (316 L) powder mix with binder 60wt% of waste polystyrene and 40wt% palm kernel oil. It is one of a critical step that must be conducted in MIM process in order to have a feedstock that is homogeneous and moldable. Water atomised Stainless Steel powder was mixed with the newly developed binder system in a Brabender Plastograph EC rotary mixer. Several tests were performed to assess the homogeneity of the feedstock that was produced at 60 vol % powder loading. The 60 vol.% was chosen because the Critical Powder Volume Concentration (CPVC) of the Powder was found to be 64.8 vol.%. The tests conducted were feedstock density, binder burn-out, rheology and SEM morphology observation. Rheological results exhibited pseudoplastic or shear thinning flow behavior, where its viscosity decreased with increasing shear rate. The feedstock viscosity also decreased with increasing temperature and was found to be suitable for molding. From all the tests conducted, it was found that the feedstock shows good homogeneity and suitable for subsequent processes in MIM.

1. Introduction

Polymer recycling is a way to reduce environmental problems caused by polymeric waste accumulation generated from daily applications of polymer materials such as packaging and construction material. It is associated with increasing awareness of environmental issues and the desire to save resources since most of these polymer materials are made from oil and gas. Currently, one of the waste plastic that widely used is expanded polystyrene. It is produced massively in order to fulfill the needs and requirement of packing industry. The total amount of plastics that ends up in waste stream is increasing parallel with the demand of this polymeric product. It is a great threat to the environment because most plastics are not biodegradable and their disposal in landfills is limited due to space limitation and its incineration is also costly. Therefore in order to address this problem, waste polystyrene could be recycled and used for the manufacturing of different valuable products to maintain the sustainability of the environment. Methods of polystyrene recycling include: Mechanical recycling which usually requires energy consumption, chemical recycling that usually requires depolymerisation and thermal catalytic recycling [1]. The present study is aimed at recycling polystyrene to act as a backbone binder in metal injection molding process.

Metal injection molding process is a manufacturing process intended to produce large amounts of small and complex metal parts. It combines the versatility and high productivity of the plastic injection molding with the powder metallurgy technique of sintering. The key points in MIM turned out to be how to make the metal flow into the mold and how to retain the shape of the molded part until it begins the sintering. This problem is commonly solved by dispersing the metal powder into a binder to
form a paste that flows at high temperature and becomes solid at room temperature. Consequently, the molded part retains its shape after injection molding and may be handled and processed safely.

Since MIM process consists of many steps, therefore characterisation of metal powder and binder components are important steps in understanding the overall process [2,3,4]. The binder acts as temporary vehicle for homogeneously packing a powder into desired shape and holding the particles until the beginning of sintering. The homogeneity of a feedstock refers to how well the particulate solid is distributed in the binder matrix. Feedstock homogeneity promote dimensional consistency of injected parts and helps preventing such defects as binder separation and powder segregation. An inhomogeneous feedstock can result in density gradients within the molded part and cause distortion. To measure the homogeneity of a feedstock several methods are available including density measurements [5], binder burnt-out [6], capillary and torque rheometry [7], and also scanning electron microscope (SEM) with back-scattered electron (BSE) imaging observation [3].

Development of new binders has always been a main interest among researchers since it led to cost and environmental issues reduction. Extensive research has been done by using natural resources binder such as Beeswax [8], Carnauba Wax [6], but fewer of them focused in waste material. The issue to be highlighted here is to evaluate the potential of using waste polystyrene as a backbone binder in MIM. The fact that the earth has tons of polystyrene disposed every day leads researcher to believe that such waste can be converted into more useful products. Therefore this study is intended to understand the feasibility of waste polystyrene (PS) as sustainable binder to produce part through MIM.

2. Experimental Procedures

2.1 Powder and Binder Characteristic

Water atomized stainless steel 316L powder having irregular shape with mean size $d_{50}$ 6 µm supplied by Epson Atmix Japan was used in this study as the metal powder. SEM micrograph of the Stainless steel powder as in figure 1 confirms the irregular shape of the metal powder. The Critical Powder Volume Concentration (CPVC) was found to be 64.8% as in figure 2. The characteristics and chemical composition of the metal powder are shown in table 1 and table 2, while table 3 shows the binder system properties. Degradation temperature of Waste PS and palm kernel oil were measured by TGA/DTA Linesis Thermo balance. It was found the highest degradation temperature is 363.4 °C and the lowest is 324.3 °C. This indicates that the mixing conditions must be below the degradation temperature of both binder constituents. At the same time, the melting temperature of Waste PS and PKO were also measured using Differential Scanning Calorimeter where the melting temperature was found to be 185.4 °C and 30 °C respectively.

![Figure 1. SEM Micrograph of SS316L at 2000x](image-url)
Table 1. Stainless Steel (316L) Powder Characteristics (Epson Atmix Japan)

| Specification   | Value                      |
|-----------------|----------------------------|
| Density         | 8.0471 g/cm³               |
| Particle Size   | D_{10} = 2.87 \mu m        |
|                 | D_{50} = 5.96 \mu m        |
|                 | D_{90} = 10.65 \mu m       |

Table 2. Chemical Composition Stainless Steel (316L) (Epson Atmix Japan)

| Element | wt%  |
|---------|------|
| C       | 0.027|
| Si      | 0.84 |
| Mn      | 0.19 |
| P       | 0.016|
| S       | 0.012|
| Ni      | 12.20|
| Cr      | 16.40|
| Mo      | 2.10 |
| Cu      | 0.03 |

Figure 2. Critical Powder Volume Concentration for SS 316L
| Binder            | Density (g/cm³) | Melting Temperature (°C) | Binder Ratio (weight %) |
|-------------------|-----------------|--------------------------|-------------------------|
| Waste PS          | 0.906           | 185.4                    | 60                      |
| Palm kernel oil   | 0.9087          | 30                       | 40                      |

2.2 Mixing Process
There are many factors that need to be considered in order to produce a homogeneous feedstock such as time, temperature, powder size and shape, formulation of binder, shear rate, and powder loading [2]. However, in this study, only mixing temperature, mixing speed and mixing time were chosen to establish a suitable mixing condition. The mixing was performed by using a rotary mixer (Brabender Plastograph EC) with rotational speed of 30 rpm. The mixing temperature was set at 190 °C, which is within the highest melting temperature (185.4 °C) and the lowest degradation temperature of the binder system (324.3 °C). This allowed complete melting of waste PS and PKO and prevented binder degradation. The polystyrene waste (PS) was fed in first followed by the addition of powder in small consecutive loadings until all the metal powder mixed evenly with melted PS. Afterward, palm kernel oil (PKO) was added into the mixer and blended with the rest of the compositions for about 60 minutes. Lastly, the blended feedstock was taken out from the mixer and left to cool at room temperature before being crushed into small pellet. Table 4 shows the calculation to produce 200 gm feedstock at 60 vol % powder loading.

| Powder | Powder | Binder | PS | PKO |
|--------|--------|--------|----|-----|
| Loading vol % | (g) | (g) | (g) | (g) |
| 60     | 186.1  | 13.9   | .34 | 5.56 |

2.3 Characterization
Homogeneity of the feedstock was analyzed by means of density measurement, using the Archimedes water immersion method according to MPIF Standard 42 [2], binder burn-out test using TGA(TGA/DTA Linesis Thermo balance and scanning electron microscope (SEM)(JSM 6380LA ) with back scattered electron imaging [3], as well rheology test using capillary rheometry (Shimadzu CFT-500D [4]).

3. Results and Discussion
The results of the density measurement for feedstock of five different samples are shown in figure 3. It is observed that there is slight difference of density value. It is due to difficulty to produce a homogenously perfect feedstock. In the binder burn-out test, the homogeneity of the feedstock was assessed by comparing the weight loss of each binder. The weight loss percentage of binder in the feedstock can be found through Thermogravimetric curves that represent the percentage of the binder in the feedstock. The results for five different samples showed the corresponding thermo gravimetric curves are better replicated and the mass change for PKO and waste PS are almost the same for each loss except slight difference which is consider minimal as shown in figure 4.
The scanning electron micrograph of the feedstock is shown in figure 5 below. Stainless steel and binder system could be distinguished as a result of various contrast levels. Stainless steel appears brighter than the binder system due to more back-scattered electrons released because of its higher atomic number. It is observed the powder particles disperse homogeneously into the matrix and are surrounded by the binder. This criterion is favorable because the homogenous feedstock of deagglomerated powders exhibits low viscosity and high flow ability.

Figure 3. Average Density (g/ cm$^3$) of Various Samples

Figure 4. Percentage Binder Mass Loss of Various Samples

Figure 5. SEM Micrograph of Feedstock at 4000x
Rheological behavior of prepared samples was assessed using a CFT-500D Shimadzu Capillary Rheometer. The samples were extruded through a die with 1 mm diameter and 10 mm length. A series of experiments were performed to investigate the rheological behavior of the binder and the prepared feedstocks. The rheological behavior of the binder was carried out at three different temperatures which are 150, 170 and 190°C as shown in figure 6. It displays the viscosity as a function of shear rate at 1000 s$^{-1}$ for the feedstock at 60% powder loading. It is clear that the viscosity of the feedstock decreases with an increasing shear rate representing pseudo-plastic rheological behavior (shear thinning) with no powder-binder separation. Summary of the important rheological properties of the feedstock is shown in table 5 below. Flow behavior index, $n$, activation energy $E$ and moldability index $\alpha$. The value of $n$ that indicates the degree of shear sensitivity should be smaller than 1 to represent the shear thinning behavior of pseudoplastic materials. Since all the $n$ values obtained from the feedstock were less than 1, it is greatly dependent on the shear rate. It is due to a low $n$ value indicates that viscosity is more dependent on shear rate. However, a very low $n$ value is undesirable because it can lead to the slip flow phenomenon that can cause molding defects. The activation energy $E$, indicates the degree of the dependence of temperature to viscosity. Since the feedstock exhibits less activation energy (less sensitivity), therefore it is suitable for injection molding. It is due, at high values of $E$, any small fluctuation in temperature and pressure during molding will result in sudden change in viscosity that will lead to stress concentration, cracks and distortion in the molded part. On the other hand, high values of $\alpha$ are desirable because the feedstock with low $\alpha$ values will be prone to powder-binder- separation. As shown in table 5, the feedstock showed high moldability indices, indicating that minimal compact defects will be produced.

### Table 5. Rheology Properties of Feedstock at Shear Rate 1000 s$^{-1}$

| Powder loading | Temp (°C) | $n$   | $E$(kJ/mol) | $\alpha_{PP}$ |
|----------------|----------|-------|-------------|----------------|
| 60%            | 150      | 0.14  | 28.3        | 1160.2         |
|                | 170      | 0.1   | 28.3        | 1202.3         |
|                | 190      | 0.17  | 28.3        | 1821.1         |

Figure 6. Rheological Test of Feedstock
Conclusion
Results of different homogeneity analysis techniques showed that the composite binder, composed of waste polystyrene and palm kernel oil can be used to produce homogenous feedstock for MIM process. It is verified by density measurement test of feedstock, where the density values are almost the same for various samples of the feedstock. Binder burn-out test also revealed the feedstock is homogenously mixed. Moreover the SEM micrograph observation also shows the powder particles dispersed homogeneously into the matrix. Rheological characterization of the feedstock also shows the feedstock possess pseudo-plastic behavior indicating no powder-binder separation. Therefore, this study confirms that waste polystyrene has the potential to be used as a binder in MIM process.

References
[1] E. Aminudin, M. F. Din, Z. Mohamad, and Z. Z. Noor, “A Review on Recycled Expanded Polystyrene Waste as Potential Thermal Reduction in Building Materials,” vol. 12, 2011.
[2] R. G. Iacocca, A critical assessment of characterization tests needed to support powder injection molding component fabrication, Review in Particulate Materials, vol. 2 (1994), 269-313.
[3] German, R. M. Powder Metallurgy & Particulate Materials Processing. Metal Powder Industries Federation, NJ. (2005).
[4] German, R.M.. Powder Injection Molding. Metal Powder Industri (MPIF). New Jersey. (1990).
[5] R. Supati, N. H. Loh, K. A. Khor, and S. B. Tor, “Mixing and characterization of feedstock for powder injection molding,” no. November, pp. 0–5, 2000.
[6] L. Liu, N. H. Loh, B. Y. Tay, S. B. Tor, Y. Murakoshi, and R. Maeda, “Mixing and characterisation of 316L stainless steel feedstock for micro powder injection molding,” Mater. Charact., vol. 54, no. 3, pp. 230–238, Mar. 2005.
[7] P. Thomas-Vielma, a. Cervera, B. Levenfeld, and a. Várez, “Production of alumina parts by powder injection molding with a binder system based on high density polyethylene,” J. Eur. Ceram. Soc., vol. 28, no. 4, pp. 763–771, Jan. 2008.
[8] K. C. Tam, S. P. Yap, M. L. Foong, and N. H. Loh, “Metal injection molding: effects of the vinyl acetate content on binder behavior,” J. Mater. Process. Technol., vol. 67, no. 1–3, pp. 120–125, May 1997.