Daftar Isi

Rheological Investigation Of Stainless Steel Powder In Bimodal Particle Size Distribution

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ABSTRACT

The rheological properties of the monomodal and bimodal MIM feedstock are presented. Stainless steel powder with particle size of 31 and 16 μm were mixed with PMMA/PEG and stearic acid to form a homogenous paste, which called feedstocks. Bimodal powders were blend at 30 to 70 % of the coarse powder distribution. Monomodal feedstock exhibits higher viscosity over the bimodal feedstocks. Binder separation is likely to occur in the monomodal feedstocks prepared with coarse powder especially at high temperature. Furthermore, bimodal feedstocks were less viscous than the monomodal but the particle size distribution had shown its influence to viscosity. The flow behavior index were decreasing when the temperature were increases. The investigation also discovered that the feedstock flow sensitivity was depends on the fine powder distributions in the feedstocks. Since all feedstocks demonstrate good pseudo plastic behavior, the feedstocks were suitable to be injection molded.

Keywords: Particle size distribution, Rheology, MIM feedstocks, flow sensitivity, bimodal.

INTRODUCTION

Metal injection molding (MIM) is a near-net shape processing technique that permits manufacturing of complex components. Fabrication starts by compounding a thermoplastic binder and powder metal mixture, referred as feedstock, followed by injection molding, binder removal and sintering [1].

This advanced manufacturing process is a modification of the common injection molding process for plastics where a significant volume fraction of plastic is replaced by a fine metal powders with a plastic binder to form a paste form feedstock, injection molding a "green" part using the specific feedstock on a conventional thermoplastic molding equipment. The major advantages from this manufacturing process include high product density, more intricate shape, higher mechanical properties, and better surface finish over traditional powder metallurgy products. Moreover, an inherent advantage of MIM is that the molding parts are hard enough to meet any needs for secondary machining [2].

The requirement for small particle dimensions has lead to some concerns regarding the potential cost of the process, making MIM a relatively expensive route for the production of larger

components. Therefore, a primary motivation for adding coarser particles to fine powders is the important cost decrease that may be achieved. However, there are some disadvantages of the method that may lead to the non-homogeneity in the sintered structure. Thus, avoidance of component defects requires a quantitative understanding of the effects of process parameters on the rheological behavior of MIM feedstocks. This is an important topic in MIM where the desire is to minimize the binder content and the sintering shrinkage using bimodal mixture [3]. Besides, broad particle size distributions or bimodal distributions are desirable to maximize the solid content, since the small particles fill interstitial space and release binder to lubricate particle flow [4]. In addition, mixtures of powders with differing sizes give improved packing densities over that available from either powder itself [3, 5, 6].

The rheological investigation described below employed bimodal powder blends with polymethyl methacrylate (PMMA), polyethylene glycol (PEG) and stearic acid as a binder system. Capillary rheometry was employed to analyze the flow behavior of the feedstocks. The information obtained from the experiment provides measurement of feedstock viscosity at variable shear rate to evaluate the feedstock stability and prediction of separation phenomena, analysis of the viscosity and shear rate to obtain the flow behavior index, activation energy and general rheological index that indicate the stability of the feedstocks as well as its suitability to the process. The bimodal particle feedstocks rheology parameters are compared with the monomodal to show the significant to the injection parameters.

METHODOLOGY

In this work, commercial gas atomized stainless steel powder 316L with particle sizes of 31 μm and 16 μm were mixed with PMMA, PEG and stearic acid. The rheological characteristic of the feedstocks were investigated using Shimadzu 500-D capillary rheometer.

Prior investigation, stainless steel powder were mixed with binders in the Sigma type blade mixer in 95 minutes at 70 °C. Four compositions of feedstocks consisting of different particles distribution were prepared for the investigation as shown in Table 1.

Table 1 Feedstock classification and the powder loading remains at 64 % V

Feedstock Abbreviation	Description
31_64	Monomodal: 31 μm
16_64	Monomodal: 16 μm
A1_64	Bimodal: 16:31 μm at 70%:30%
B1_64	Bimodal: 16:31 μm at 30%:70%

RESULTS AND DISCUSSION

Viscosity dependence to shear rate

Figures 1, 2 and 3 show the correlation of viscosity and shear rate at injection temperatures of 120 °C, 130 °C, and 140 °C. These temperatures were selected based on literature [7], which studies the injection parameters of the MIM feedstock prepared with the same materials as presented by this paper.
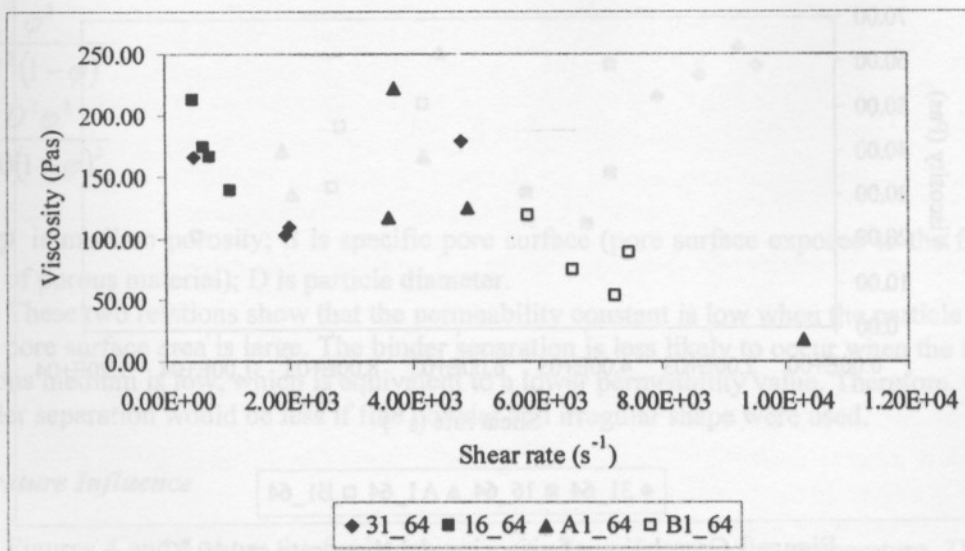
Figure 1 Correlation of viscosity and shear rate at 120 °C

The bimodal feedstock, A1_64 as shown in Figure 1 exhibits highest viscosity while B1_64 is the lowest at this injection temperature. Powder-binder separations occur at high shear rate on the monomodal feedstocks, 31_64 and B1_64. However, when the temperature was increased to 130 °C (Figure 2) the viscosity of A1_64 is reducing.
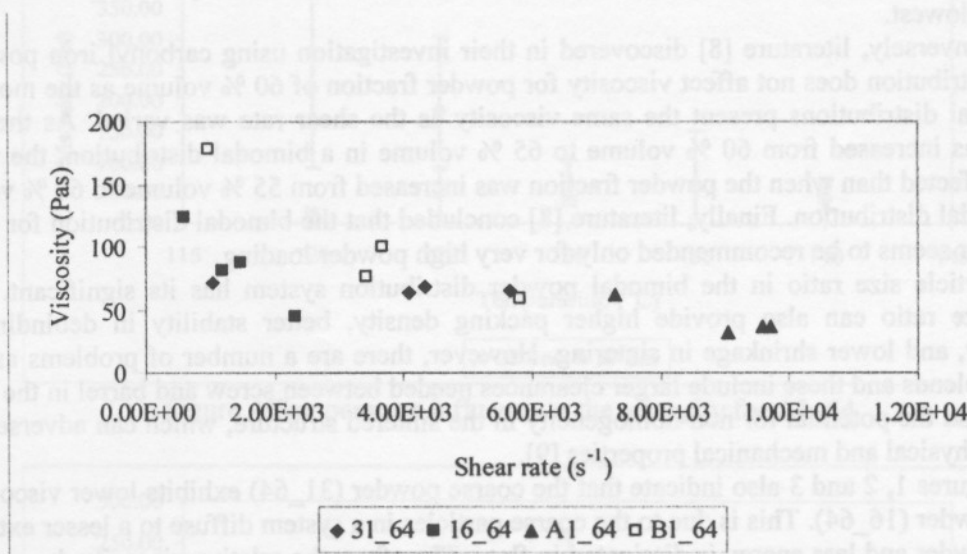

Figure 2 Correlation of viscosity and shear rate at 130 °C

When the injection temperature was increased to 130°C, the bimodal feedstock, B1_64 exhibits higher viscosity at shear rate less than 2000 s⁻¹ compared to the monomodal feedstocks. However, at shear rate lower than 8000 s⁻¹, A1_64 is more viscous compared to the B1_64. However, the viscosity is almost the same at high shear rate.
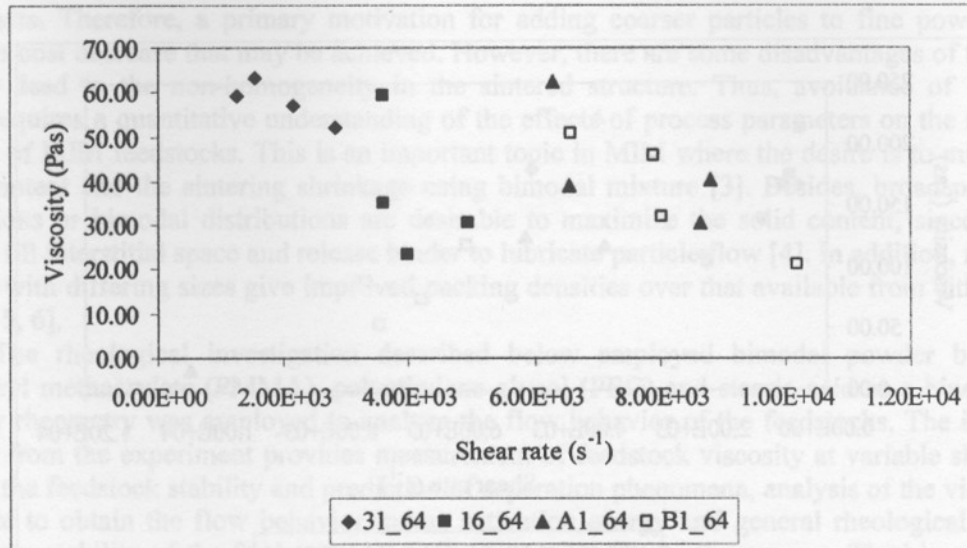
Figure 3 Correlation of viscosity and shear rate at 140 °C

Moreover, when the temperature was increased to the maximum (Figure 3), the monomodal feedstock, 31_64 became more viscous than 16_64. This is possibly due to binder separation of the coarse powder. At the mean time, the bimodal feedstock A1_64 is more viscous than B1_64. However, the bimodal feedstocks were still dominant in term of viscosity compared to the monomodal feedstocks in every injection temperatures. The bimodal particles distribution does affect the feedstock viscosity where as seen in Figures 1, 2 and 3 that the viscosity of A1_64 and B1_64 is among the lowest.

Conversely, literature [8] discovered in their investigation using carbonyl iron powder that particle distribution does not affect viscosity for powder fraction of 60 % volume as the monomodal and bimodal distributions present the same viscosity as the shear rate was varied. As the powder fraction was increased from 60 % volume to 65 % volume in a bimodal distribution, the viscosity was less affected than when the powder fraction was increased from 55 % volume to 60 % volume in a monomodal distribution. Finally, literature [8] concluded that the bimodal distribution for carbonyl iron powder seems to be recommended only for very high powder loading.

Particle size ratio in the bimodal powder distribution system has its significant. A large particle size ratio can also provide higher packing density, better stability in debinding, good moldability, and lower shrinkage in sintering. However, there are a number of problems associated with such blends and these include larger clearances needed between screw and barrel in the molding machine, and the potential for non-homogeneity in the sintered structure, which can adversely affect the part's physical and mechanical properties [9].

Figures 1, 2 and 3 also indicate that the coarse powder (31_64) exhibits lower viscosity than the fine powder (16_64). This is due to the coarse particles in a system diffuse to a lesser extend than the fine powder and less energy is dissipated in flow. Therefore, the relative viscosity decreases with the increase of the particle size. Consequently, by adding coarse powder to the fine powder, feedstock with lower relative viscosity at the same solid loading content can be obtained [9].

Binder separation phenomena are likely to occur in the 31_64 at all temperatures. Nevertheless, Figures 1, 2 and 3 does not indicate any occurrence of binder separation phenomenon in 16_64. Modelling the binder separation phenomenon as flow through porous medium enables identification of the parameters influencing the separation that occurred during the tests. The following equations (Kozeny-Carman and the Blake Kozeny equation respectively) describe the permeability constant of a porous medium, k

$$k = \frac{\varphi^3}{5S^2(1-\varphi)^2} \qquad (1a)$$

$$k = \frac{D^2\varphi^3}{150(1-\varphi)^2} \qquad (1b)$$

where φ is medium porosity; S is specific pore surface (pore surface exposed to the fluid per unit volume of porous material); D is particle diameter.

These two relations show that the permeability constant is low when the particle size is small and the pore surface area is large. The binder separation is less likely to occur when the flow through the porous medium is low, which is equivalent to a lower permeability value. Therefore, the tendency for binder separation would be less if fine powder and irregular shape were used.

Temperature Influence

Figures 4 and 5 show the feedstock viscosity dependence to the temperature. The error bars indicates the maximum and minimum viscosities. As shown in Figure 4, A1_64 has broad viscosity range at temperature 120 and 125 °C, when it was extruded at 29 bars from the rheology test barrel. Besides, in Figure 5, B1_64 also shows wide viscosity range at 130 °C when pressures at 29 and 59 bars were applied.
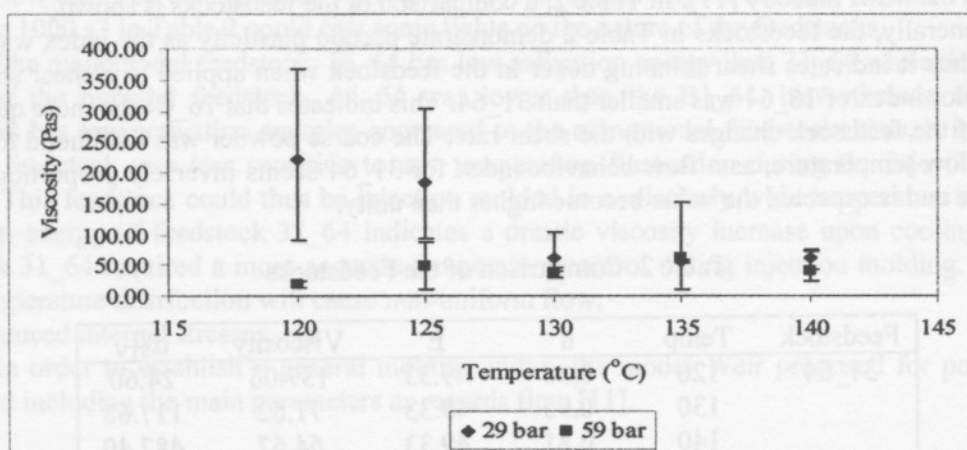

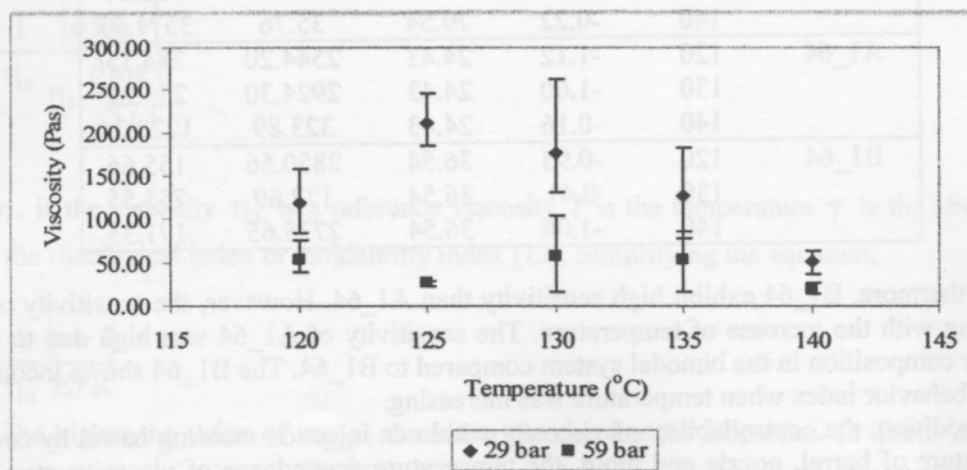Figure 4 Temperature influence to the viscosity for A1_64

Figure 5 Temperature influence to the viscosity for B1_64

In Figure 4, the viscosity was decreased when the extrusion pressure and the test temperature was increased. Regardless, in Figure 5 the viscosity was fluctuates with the increasing of temperatures. This is possibly due to the occurrence of binder separation as the feedstock in Figure 5 has broader coarse powder distribution compared to A1_64.

Feedstock Pseudo Plasticity

A regression line of the scatter plot shown in Figures 1, 2 and 3 could be rewritten as

$$\eta = K \dot{\gamma}^{n-1} \qquad (2)$$

where η is the viscosity at shear rate $\dot{\gamma}$, K is a constant and, n is a flow behavior index. Equation (2) has been widely used to correlate the data of viscosity to shear rate for pseudo plastic and dilatant fluids, which is known as the power-law equation.

The flow behavior index, n of the power-law index indicates the shear sensitivity. Smaller n of feedstock indicates higher shear sensitivity and more pseudo plasticity of the feedstocks. Some molding defects such as jetting are associated with small n, i.e., higher shear sensitivity [10]. During the injection molding process, pseudo plastic behavior is desirable and, therefore, a decrease in viscosity with an increase in the shear rate. This dependent behavior of the viscosity against the shear rate is especially important when producing complex and delicate parts, which are important products in the MIM industry [11]. In Table 2, a comparison of the feedstocks is shown.

Generally, the feedstocks in Table 2 demonstrate pseudo plasticity as the index was smaller than one, thus it indicates shear thinning occur in the feedstock when applied to a shear stress. The flow behavior index of 16_64 was smaller than 31_64. This indicates that 16_64 the more quickly the viscosity of the feedstock changes with the shear rate. The coarse powder was likelihood to become dilatant at low temperature, as a flow behavior index for 31_64 seems inversely proportional to the temperature and is expected the value become higher than unity.

Table 2. Comparison of the Feedstocks

Feedstock	Temp	n	E	Viscosity	α_{STV}
31_64	120	0.98	49.33	137.06	24.60
	130	0.95	49.33	71.62	117.68
	140	0.81	49.33	64.67	487.40
16_64	120	0.49	79.54	141.77	376.03
	130	0.23	79.54	94.44	852.24
	140	-0.22	79.54	35.76	3571.93
A1_64	120	-1.12	24.43	2544.20	284.15
	130	-1.00	24.43	2924.30	233.23
	140	-0.16	24.43	323.89	1222.24
B1_64	120	-0.95	36.54	2850.56	155.66
	130	0.41	36.54	177.69	755.55
	140	-1.06	36.54	2735.65	171.35

Furthermore, B1_64 exhibit high sensitivity than A1_64. However, the sensitivity of B1_64 was reducing with the increase of temperature. The sensitivity of A1_64 was high due to the high fine powder composition in the bimodal system compared to B1_64. The B1_64 shows inconsistency of the flow behavior index when temperature was increasing.

In addition, the controllability of viscosity within an injection molding barrel by controlling the temperature of barrel, nozzle and mold, the temperature dependence of viscosity may have an effect on the response of the material to the sudden non-uniform cooling within cavity. For example,

during the molding stage, the feedstock was forced into the mold where it immediately began to cool. If the cooling was accompanied by a rapid increase in the viscosity, the result may be incomplete filling the mold and introduced cracking or porosity in molded parts. Therefore, low temperature dependence was desired to minimize problems arising from fluctuating molding temperatures, thereby minimizing stress concentration, cracks and shape distortion [4, 12]. The value of flow activation energy, E as shown in Table 2 represent the influence of temperature on the viscosity of the feedstocks, is an important parameter for injection molding

$$\eta = \eta_0 \exp\left(\frac{E}{RT}\right)$$ (3)

where R is the gas constant, T the temperature and η_0 the reference viscosity.

Taking nature logarithm at both sides, equation (4) was obtained

$$\ln \eta = \ln \eta_0 + \left(\frac{E}{R}\right)\frac{1}{T}$$ (4)

With a shear rate of 1000 s^{-1}, which fell in the normal range of shear rates for injection molding of MIM feedstocks, by plotting $\ln \eta$ against the reciprocal temperature, the activation energies of viscous flow could be calculated and given in Table 2. The activation energies and the viscosities at shear rate 1000 s^{-1} in Table 2 could cast some lights on the nature of the feedstocks.

The monomodal feedstock, 16_64 has low activation energy than 31_64 while the activation energy of the bimodal feedstock, A1_64 was lower than the B1_64. Nevertheless, the bimodal feedstocks has low activation energies compared to the monomodal feedstocks thus, it indicates the bimodal feedstock was less sensitive to any temperature fluctuation during the injection molding process. This feedstock could thus be injection molded in a relatively wide temperature range. High activation energy of feedstock 31_64 indicates a drastic viscosity increase upon cooling, and thus feedstock 31_64 required a more accurate temperature control during injection molding. Otherwise, mold temperature distribution will cause non-uniform flow, which induced internal stresses.

In order to establish a general molding index, the model Weir proposed for polymers has been used including the main parameters as regards flow [11].

$$\alpha_{STV} = \frac{1}{\eta_0} \frac{\left|\frac{\partial \log \eta}{\partial \log \dot{\gamma}}\right|}{\eta_0 \frac{\partial \log \eta}{\partial 1/T}}$$ (5)

where, η, is the viscosity η_0 is a reference viscosity T is the temperature $\dot{\gamma}$ is the shear rate and α_{STV} is the rheological index or moldability index [13]. Simplifying the equation:

$$\alpha_{STV} = \frac{1}{\eta_0} \frac{|n-1|}{E/R}$$ (6)

The higher the value of α_{STV}, the better the rheological properties. In Table 2, 16_64 has better rheological properties at 140 °C while 31_64 has poor rheological property at 120 °C. In general, the rheological properties are proportional to the temperatures.

CONCLUSIONS

The rheological behavior of monomodal and bimodal stainless steel MIM feedstock has been investigated in terms of injection temperature (120-140 °C) and particle size distributions, over wide range of shear rate.

All feedstocks are possible to be injection molded as the flow behavior index indicates pseudo plastic behavior. The bimodal particles distribution enables to increase the shear rate and thus reduce the feedstock viscosity. B1_64 is less viscous compared to the A1_64. In addition, the B1_64 particles distribution was organize according to the Furnas model, which describes the ideal particle packing behavior of a binary powder system [13].

Furnas model suggested that the volume of fine particles is between 30 to 50 %. The bimodal feedstock also exhibit better pseudo plasticity compared to the monomodal feedstock. Further, the sensitivity of the viscosity to the temperature is also small thus; the bimodal feedstock is stable over wide range of temperature.

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