

# RHEOLOGICAL PROPERTIES OF SS316L MIM FEEDSTOCK PREPARED WITH DIFFERENT PARTICLE SIZES AND POWDER LOADINGS

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## ABSTRACT

Rheological study has been performed experimentally to evaluate the influence of different particle sizes and powder loadings to the rheological properties of SS316L MIM feedstock. The effects of different particle size, powder loading, shear rate and temperature has been investigated using capillary rheometer. The effect of shear rate to the feedstock viscosity was evaluated and result demonstrated that the feedstocks are pseudo-plastic. Their viscosity also shows strong dependence on the shear rate. The activation energy of each feedstock indicates the sensitivity to temperature and shear stress. The moldability index of the feedstock is also significant. The results indicate that the high powder loading feedstock at 64% volumes demonstrate the best rheological properties. The feedstock achieved desirable injection molding characteristics, such as homogeneous, stable, flow behavior index,  $n < 1$ , moderate activation energy ( $E$ ) and high moldability index ( $\alpha$ ).

**Keywords:** Metal Injection Molding (MIM), Particle Size, Powder Loading, Rheological Properties

## 1.0 INTRODUCTION

Metal Injection Molding (MIM) is a manufacturing process capable of producing high-performance and complex shaped metal parts at low cost [1, 2]. In general, MIM is the process of mixing fine metal powders with plastic binder to form a feedstock, injection molding of the green part, extracting the binder, and finally sintering. The major advantages of this process includes high sintered density, more intricate shape, better mechanical properties, and better surface finish compared to the traditional powder metallurgy compacts [3]. Recent interest had been directed at particular MIM components with high added value, including sport equipments, eyeglasses, wristwatches, jewellery, and *etc.* [2].

The molding stage is the critical step for fabricating compact parts without cracking or distortions. This step requires specific rheological behavior of the feedstock such as flow behavior index,  $n < 1$  and moderate amount of activation energy,  $E$ . Non-homogeneous melt flow and powder-binder separation during the injection molding process may defects the compacts, resulting in cracking and warpage during debinding and sintering, and ultimately poor physical and mechanical properties of the final parts [2]. Rheological analysis can be made to quantify the

stability of the feedstock for achieving a successful manufacturing process [4, 5, 6].

The most important rheological property in MIM is viscosity, which relates a shear stress to shear rate. It is known that, a molded part would obtain its desired shape without any physical defects when the viscosity is controlled within a narrow range [7]. High melt viscosity has been identified as a reason for the molding difficulty [4, 5]. The melt viscosity is influenced by the particle size distribution, particle shape and the density of the powder, and it can be reduced by adding additives such as lubricants and plasticisers [8]. It is known that feedstock viscosity with low temperature sensitivity and low sensitivity to shear thinning behavior is desired [7]. Thus, parameters such as flow behavior indexes,  $n$ ; activation energy,  $E$ ; and moldability index,  $\alpha$ ; are important in the rheological investigations.

Most experiments that investigate the rheological properties of the MIM feedstocks are focusing on the best rheological properties. Huang *et al.* [3] investigates on the effects of shear rate, solid volume fraction (powder loading) and melt temperature on the rheological behavior of the Fe/Ni MIM feedstocks, prepared by readily available polymer binder systems. He concluded that the binder system which had stable relationships in both the shear-rate dependence and temperature-dependence

of the viscosity would be selected as a binder for the feedstock. Furthermore, Karatas *et al.* [4] studied the rheological properties of the ceramic feedstocks, using polyethylene (PE) and three waxes (carnauba, bees wax and paraffin) with steatite powder. The experiment concluded that the formulation proposed has met the specific requirement and suitable to be injection molded. The flow behavior index ( $n$ ) parameters have been determined to be less than 1, indicated a pseudo-plastic behavior.

For evaluating the influence of TiC addition on the rheological behavior and stability of SS316L MIM feedstock, Khakbiz *et al.* [7] performed a rheological study using a capillary rheometer. They concluded that the rheological behavior of the feedstocks is highly depends on the blend composition. The addition of TiC particles to the stainless steel powder enhances the feedstock viscosity at relatively low shear rates, *i.e.*  $< 500\text{s}^{-1}$ . Furthermore, the feedstock instability increases, particularly at higher powder loading. By increasing shear rate and temperature, the viscosity decreases and the instability of the feedstock has improved. Krauss *et al.* [6] conducted an experiment using capillary rheometer to analyse the rheological behavior of alumina powder feedstock containing polyethylene glycol (PEG), polyvinylbutyral (PVB) and stearic acid (SA) at different powder loadings. Some of the feedstocks showed a pseudo-plastic behavior ( $n < 0$ ) and the viscosity displayed strong dependence to the shear rate. The results indicate that the low powder loading feedstock exhibits a best rheological behavior. Meanwhile, an experiment performed by Faiz Ahmad [9] to study the flow properties of composite mixes comprised of aluminum powder and glass fibers compounded into a plastic binder. The optimum powder loading was determined and the viscosity of the composite mixture decreased with the increase of shear-rate. The optimum level of fiber content would reduce the relative viscosity of the composite mixture. Composite mixture with longer fibers results in high viscosity. This present paper evaluates the effect of powder loadings and particle sizes to the rheological behavior and moldability of SS316L feedstock. This involves an investigation to the influence of temperature and shear rate to the viscosity of the MIM feedstocks.

## 2.0 EXPERIMENTAL PROCEDURES

### 2.1 Starting Materials

The metal powder used in the study is a gas atomised SS316L powder (ANVAL, Sweden). The characteristic of the powder is as shown in Table 1, while Table 2 shows the chemical composition of the powder.

**Table 1: Characteristics of metal powder used in this study**

Powder	Stainless steel, SS 316L
Manufacturer	ANVAL, Sweden
Particle shape	Spherical
$D_{50}$	11.225 $\mu\text{m}$ (fine) and 19.606 $\mu\text{m}$ (coarse)
Pycnometer density	7930 $\text{kg}/\text{m}^3$

**Table 2: Chemical composition of the metal powder**

C	Si	Mn	P	S
0.027	0.44	1.39	0.026	0.01
Cr	Ni	Mo	Fe	
16.7	10.4	2.09	balanced	

The binder system composed of polyethylene glycol, PEG as the major component (73%), polymethyl methacrylate, PMMA as backbone polymer (25%), and stearic acid as surfactant (2%). The surfactant acts as lubricant that enhances the dispersion of powder in the binder during mixing, and also enhances the powder loading and green strength [8] without sacrificing the flow properties of the mixtures.

### 2.2 Compounding

In order to study the effect of powder loading and particle size to the rheological behavior of the MIM feedstocks, the formulations of coarse and fine powder feedstock with powder loading of 62 and 64% volume is prepared. The remaining volume fraction is binder, consists of 73% weight of polyethylene glycol (PEG) and 25% weight of polymethyl methacrylate (PMMA). About 2% weight of stearic acid (SA) is used as a surfactant.

**Table 3: Formulation of feedstocks**

Powder Loading (% Volume)	Powder particle	Abbreviation
62	Fine	F62_16
62	Coarse	F62_31
64	Fine	F64_16
64	Coarse	F64_31

Table 3 illustrates the composition of the feedstock used in this study. The PMMA powder was dissolved with acetone for 15 minutes at 1 *gm.* of PMMA for each 4 *ml* acetone. The feedstock was prepared with Z-blade type mixer, having a rotation frequency of 25 *rpm.* The components of the feedstock were added simultaneously at room temperature for 30 minutes. The mixer temperature was set at 70°C and the mixing continued until 1 hour. After mixing, the dough is removed from the mixer and cooled to 60°C, before the dough is subsequently fed into a strong crusher in order to produce homogenised granules.

### 2.3 Rheological Properties Measurement

The test was conducted using a CFT-500D Shimadzu capillary rheometer as shown in Figure 1. L/D=10 die was attached underneath the extruder barrel. The test was conducted at constant capillary temperature of 125°C and the load applied to the tester ranges from 30-60 *kgf.*

The barrel was filled with the feedstock and pressed lightly using a piston before it left in the barrel for 10 to 15 minutes to

attain a thermal equilibrium. The pressure drop across the die was recorded for calculating the shear stress at the die wall. The flow rate through the capillary was calculated using a relation provided by the Japanese Industrial Standard, JIS K7210 [10]:

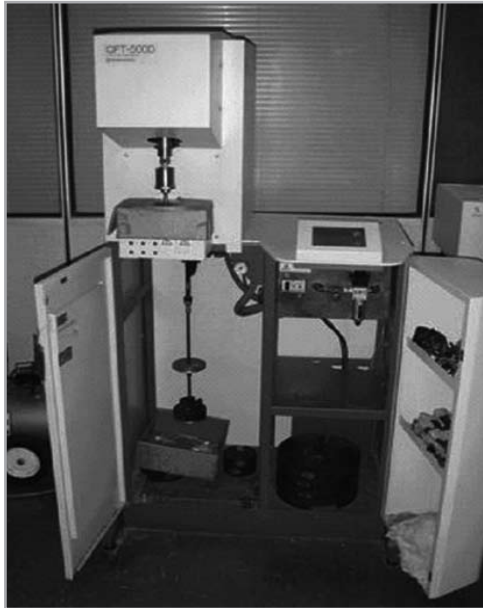


Figure 1: CFT-500D Shimadzu capillary rheometer

$$Q = 0.4/t \text{ (cm}^3\text{/s)} \tag{1}$$

Where,  $t$  is the time for the piston to travel from 3mm point to 7mm point in the barrel. The shear rate  $\dot{\gamma}$  was calculated using Equation (2):

$$\dot{\gamma} = 32/\pi D^3 * 10^3 \text{ (s}^{-1}\text{)} \tag{2}$$

Where,  $D$  is the capillary diameter, 1mm. The flow behavior index is determined by the Power law equation as shown in equation (3):

$$\eta = K \dot{\gamma}^{n-1} \tag{3}$$

Where,  $\eta$  is the viscosity,  $K$  is the constant. While, the Arrhenius's equation is applied to determine the activation energy,  $E$  as shown in Equation (4):

$$\eta = \eta_0 \exp(E/RT) \tag{4}$$

Where  $\eta_0$  is the viscosity at reference temperature,  $R$  is the gas constant and  $T$  is the melt temperature. According to equation (4),  $\ln \eta$  vs.  $1/T$  at certain shear rate is obtained. The graphs show tendency to fit into straight lines. The slopes of the graph indicate that the temperature seems to be dependent to the viscosity, or the activation energy,  $E$ . Large values of the activation energy indicate high sensitivity of viscosity to temperature.

The moldability index ( $\alpha$ ) of the binder formulations and PIM feedstock is calculated using Equation (5) proposed by Weir *et al.* [4, 5]:

$$\alpha = 10^9 (n)/\eta_0 (E/R) \tag{5}$$

Where,  $\eta_0$  is the apparent viscosity at the reference shear rate at 100, 10 000 and 100 000  $s^{-1}$ .

### 3.0 RESULTS AND DISCUSSION

#### 3.1 Flow Behavior Index, $n$ (Shear Sensitivity)

Figure 2 shows the viscosity of the feedstock as a function of shear rate at 125°C. The melt exhibit a shear thinning or pseudo-plastic behavior. The viscosity of a pseudo-plastic substance decreases as the shear rate increases (shear thinning). This could be due to particle orientation and ordering with flow as well as breakage of particle agglomerates released together with the binder [2].

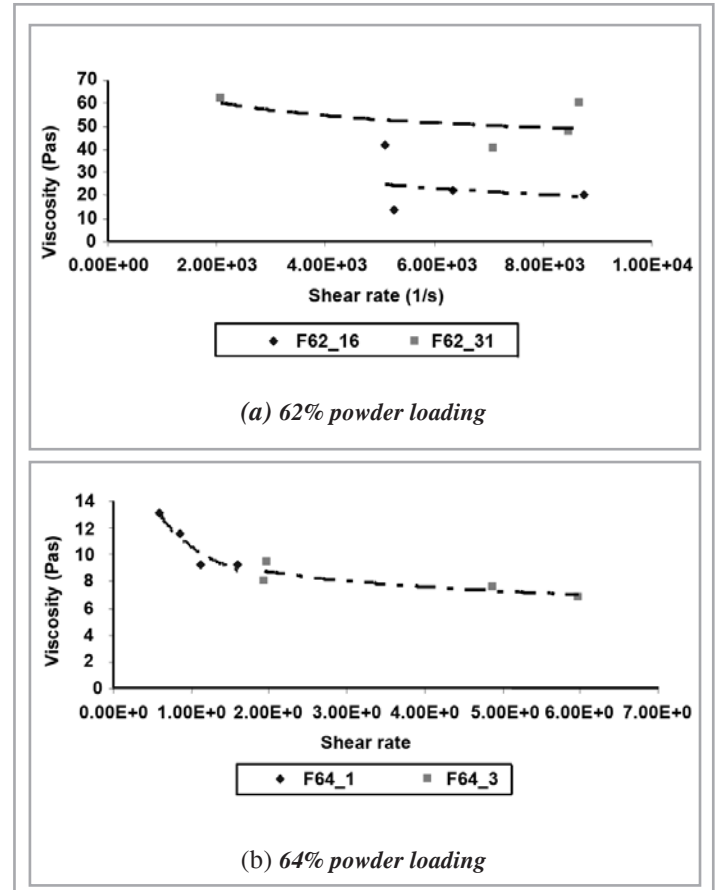


Figure 2: Viscosity of MIM feedstock as a function of shear rate at temperature of 125°C. (a) 62% powder loading and (b) 64% powder loading

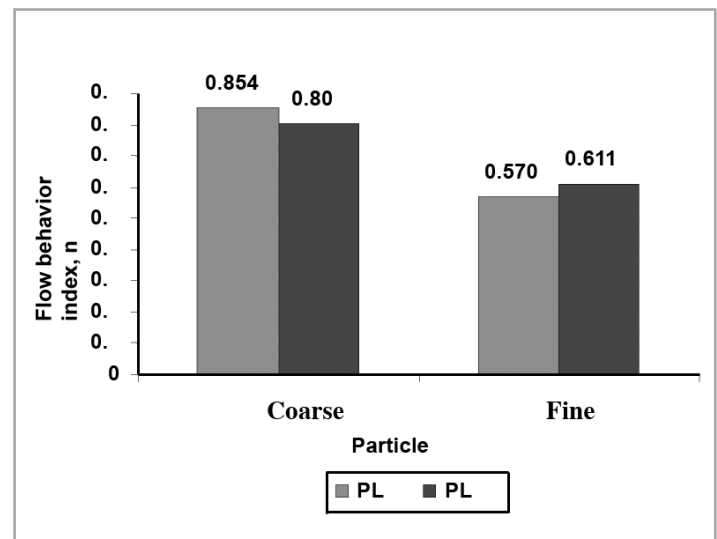


Figure 3: The flow behavior index of feedstock at 125°C as a function of particle size at different powder loading

Figure 3 shows the flow behavior index,  $n$  of the feedstock at 125°C. Most feedstock exhibit a shear thinning or pseudo-plastic behavior, as the flow behavior index,  $n$  is smaller than 1. The lower the value of the flow behavior index, the more viscosity dependence to the shear rate [7]. However, some molding defects such as jetting is associated with low flow behavior index. Jetting is undesirable since it causes severe defects, including weld lines and other imperfections in the final molded part [6]. For coarse particle feedstock, the flow behavior index decreases as the powder loading increases; this is in contrast to the fine powder feedstock. This means that the viscosity is more sensitive to shear rate particularly for the coarse powder feedstock. Feedstock F62\_16 has the lowest value of flow behavior index compared to other feedstock, indicates that the feedstock is much dependence to the shear rate and demonstrates the best pseudo-plastic behavior. But this is undesirable because lower the flow behavior index is leading to the slip flow phenomena, which cause molding defects. Thus F62\_31 is considered as the best feedstock in term of flow behavior index.

### 3.2 Activation Energy, $E$ (Temperature Sensitivity)

Another important characteristic of MIM feedstock is temperature-dependence to the viscosity. Pure binder has a viscosity that usually varies exponentially with absolute temperature  $T$  [2]. Therefore, the effect of temperature on the viscosity of the feedstock was evaluated.

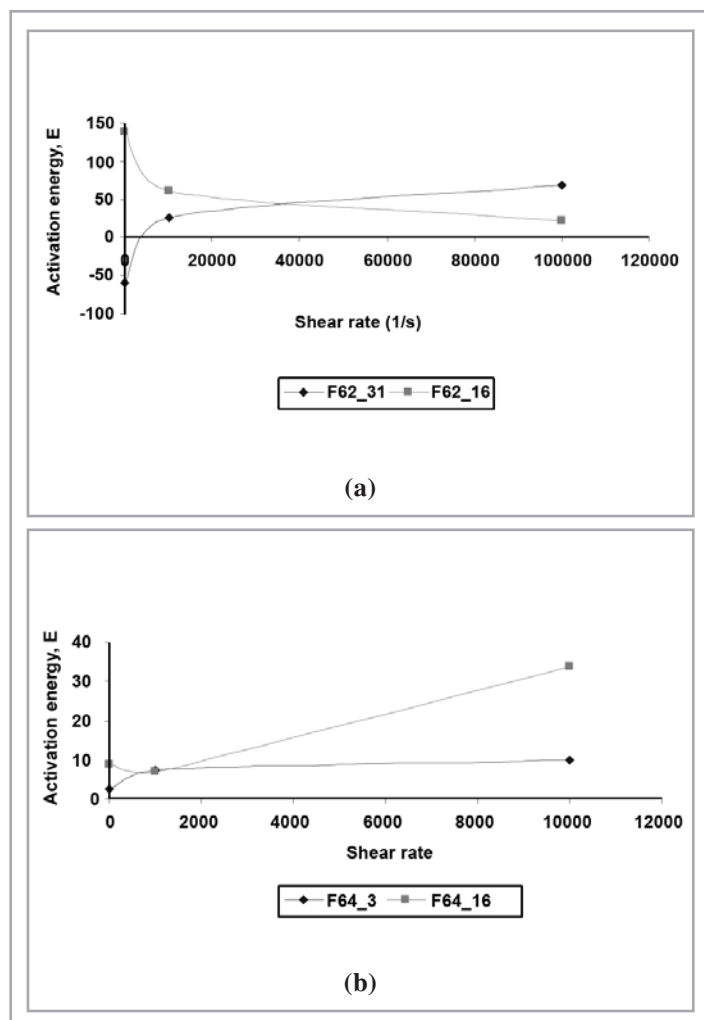


Figure 4: Activation energy of PIM feedstock as a function of shear rate at different powder loadings of (a) 62% and (b) 64%

The activation energy as a function of shear rate is shown in Figure 4. The activation energy of the coarse powder feedstock is proportional to the shear rate. Furthermore, at higher powder loading, the activation energy was found to be high, particularly at high shear rates. High value of flow activation energy indicates a strong temperature-dependence of the feedstock to the viscosity. Therefore, any small fluctuation of temperature during molding results in a sudden viscosity change, resulting defects in the molded parts, such as cracking and distortion due to the stress concentration [7]. In addition, feedstock with high temperature sensitivity is also sensitive to pressure [2]. As pressure increases, the melt is becoming more viscous. But in contrast the fine powder feedstock demonstrates the inverse proportional of the activation energy to the shear rate, particularly at low shear rates. The small value of the activation energy result less temperature sensitive, thus it minimizes stress concentration, cracks and distortion on the molded parts. Figure 4 exhibits the flow activation energy is high at high powder loading. The occurrence of a powder-binder separation phenomenon during a rheological testing is the possible reason for the increase of the activation energy.

### 3.3 Moldability Index, $\alpha$

The feedstock moldability index is shown in Table 4. The index has been calculated using Equation (5).

Table 4: Moldability index,  $\alpha$  obtained in this study

Feedstock	Moldability index, $\alpha$
64_31	-5.07E+03
62_31	-6.41E+04
64_16	-4.28E+03
62_16	-4.97E+03

Table 4 demonstrates that the moldability index is for the coarse and fine powder feedstock decreases as the powder loading increases. This result is in good agreement with Khakbiz [7], that the higher powder loading led to the high moldability index. In the absence of problems such as jetting or high residual stresses, the higher value of the moldability index is desirable since feedstock with low flow behavior index are prone to powder-binder separation. As shown in Table 4, the F64\_16 exhibits the highest value of the moldability index and thus is the best feedstock to be injection molded.

## 4.0 CONCLUSIONS

The rheological behavior of the feedstocks composed of 316L stainless steel has been investigated. The results revealed the pseudo-plastic or a shear-thinning behavior of the feedstock. Clearly, the fine the particle size, the lower the flow behavior index and thus indicates greater pseudo-plastic behavior of the feedstock. The high sensitivity of shear thinning is undesirable because it leads to the slip flow phenomenon, causes a molding defects. Coarse powder and fine powder feedstock at low and high powder loading respectively is more temperature-dependence to the viscosity (higher value of activation energy). This indicates less temperature tolerance during injection molding since any

small fluctuation of temperature during molding results in a sudden viscosity change. Meanwhile, the value of the moldability index,  $\alpha$  decrease as the powder loading increased, and is same for both particle sizes. High value of the moldability index is desirable since feedstock with low value of flow behavior index prone to powder-binder separation. A feedstock with a good homogeneity, high stability, a high flow behavior index, low activation energy and high moldability index were found to have optimum injection molding characteristics. Thus, 64% volume is identified as the best powder loading for the injection molding. ■

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