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RHEOLOGICAL INVESTIGATION OF WATER ATOMIZED METAL INJECTION MOLDING (MIM) FEEDSTOCK FOR PROCESSIBILITY PREDICTION

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Abstract. This paper presents the rheological properties of SS316L water atomized MIM feedstock. Coarse and fine SS316L water atomized powder is mixed with a composite binder consisting of PMMA and PEG to form a homogenous paste, termed as feedstock. The feedstock is loaded with SS316L water atomized powder ranging 62 v/o, 62.5 v/o, 63 v/o, 63.5 v/o and 64 v/o. However, due to the morphology of the water atomized powder which is not spherical compared to the gas atomized ones, fine powder feedstock is unable to produce any significant rheological result due to the powder loading being more than 63.5 v/o. Results show that the fine powder feedstock demonstrates a higher viscosity if compared to the coarse powder feedstock. It can be established that binder separations are likely to occur in the coarse powder feedstock, especially, at high temperatures. The investigation concludes that the fine powder feedstock has its best rheological properties at 62 v/o while the coarse powder feedstock lies between 63 v/o and 63.5 v/o.

1. INTRODUCTION

Metal injection molding (MIM) is an alternative method for production of small, irregular shaped parts that have fine tolerances, good surface finishes and are low in prices [1,2]. MIM is the main competitor of investment casting, especially for the production of small parts in large quantities [3, 4]. In this method, metal powders are mixed with a suitable binder (commonly a polymer) before it is granulated to form a feedstock. The feedstock is first fed into an injection machine and then injected into the mold cavity by pressure, as been done for plastic material. After the green part has been ejected from the mold cavity, the binder is removed by dissolving it in a solvent or by thermal or chemical decomposition. Finally, the sintering of MIM parts (identical to that of classical powder metallurgy) is required in order to provide the product with a tough property.

Furthermore, for the reason that gas atomized powder feedstock is easy for injection molding, it is therefore very common for producing relatively small and complex components with no subsequent processes [5, 6, 7, 8, 9]. This is due to the fact that the gas atomized powders are spherical. However, water atomized powders are rounded and ligamental in shape, producing a lower tap density. In contrast, gas atomized powders have lower oxygen contents and exhibit low mixture viscosities in molding [1].

The advantage of the water atomized powder lies in its low cost. Also, the brown strength of parts produced with this kind of powder is fairly high as the irregular particles cannot flow past each other. Nevertheless, it has its disadvantages such as low tap density resulting in high sintering shrinkage as well as the tendency of the irregular particles to slightly align during injection molding and thus causing anisotropic shrinkage [10].

The rheological properties are important in the injection molding process, since they involve the flow of the molten feedstock into the mold cavity. Rheological analysis can be made to quantify the stability of the feedstock during molding process [11].

This study focuses on the rheological properties of the coarse and fine SS316L water atomized powder feedstock based on composite binder consisting of polyethylene glycol (PEG) as a primary binder while polymethyl methacrylate (PMMA) as the secondary binder and stearic acid as a surfactant with the final goal of evaluating the suitability of these feedstocks for use in injection molding. This comes from the fact that, the feedstock fed into the injection machine should be injected into a mold at an appropriate temperature and pressure. A low injection temperature results in high flow viscosity and causes die filling problems. On the other hand, a high temperature results in the separation of the powder and the binder, and also causes high shrinkage of the final product. The injection pressure will have similar effects which are: low pressure results in a die filling problem and high pressure results in binder separation. It is important to know the volume percentage of the powder and the binder in the feedstock before putting it into the injection machine. If the volume percentage of the powder is high, the injection process will be difficult. Excessive binders, on the other hand, will result in high shrinkage of the final product.

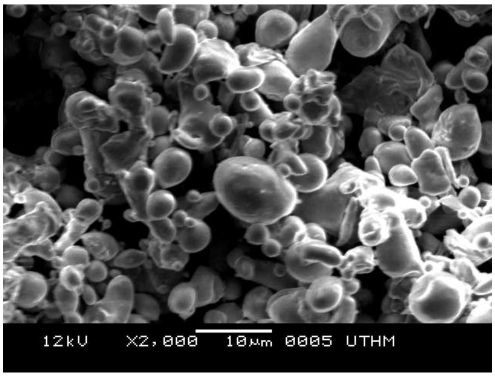
2. METHODOLOGY

2.1 Materials

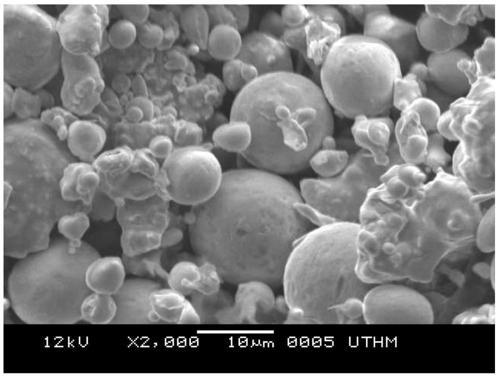
Metal powder used in this study is SS316L water atomized powder supplied by Epson Atmix Corporation, Japan. The SEM image and particle size analysis of the powder is as shown in Fig. 1 and Table 1 correspondingly.

With reference to Fig. 1, note that the water atomized SS316L powder used in this investigation is ligament shaped, but generally its shape is almost spherical as in the gas atomized powder which is predominantly used for MIM. The particle size analysis shown in Table 1 was conducted using Malvern Mastersizer and the D_{50} represents a mean diameter of a powder particle.

In order to help SS316L powder to flow easily into the mold cavity, polymer based binder is used to bind the powder particles together. Hence, the binder will act as a temporary vehicle for homogenously packing a powder into a desired shape and therefore hold the particles in that shape from the beginning of sintering. Although the binder should not dictate the final composition of molded material, it has a major influence on the success of MIM processing [1]. The major goal of the binder system is to provide the necessary flowability and to enable filling of a cavity during the injection molding process. The binder is subsequently removed from the mixture during debinding and the first stage of the sintering process [12]. The volumetric percentage (v/o) of powder in the mixture is termed as “powder loading” of the feedstock. This value has a large effect on virtually all properties of the feedstock.



(a). SEM of the fine powder



(b). SEM of the coarse powder

Fig. 1 SEM photograph of ATMIX SS316L water atomized powder

Table 1 Particle size (μm)

	D_{10}	D_{50}	D_{90}
Coarse	4.968	15.059	34.753
Fine	3.338	7.148	17.192

Consequently, a binder system based on polyethylene glycol (PEG) is used in the investigation. The minor component is polymethyl methacrylate (PMMA) and, stearic acid (SA) is added as the surface-active agent. The binder composition consists of 73 % PEG + 25 % PMMA + 2 % SA based on the weight fraction.

2.2 Experiment Procedure

In a previous investigation, SS316L powders were mixed with binders in a sigma blade mixer for 190 minutes at 70 ^{o}C. After mixing, the paste was removed from the mixer and was fed into a strong crusher for granulation. The rheological characteristic of the feedstock was investigated using Shimadzu 500-D capillary rheometer with a die of L/D = 10.

3. RESULTS AND DISCUSSION

Fig. 2 and 3 demonstrate the apparent viscosity of the fine and coarse powder feedstock respectively at 150 ^{o}C. Both figures show the typical shear rate, thinning effect known for polymers which proves that an increasing shear rate decreases the viscosity. Fig. 2 shows that the viscosities and the shear rate effect are strongest for fine powder feedstock at powder loading of 62 v/o if compared to another powder loading of the same feedstock. As shown in Fig. 2, the melt viscosities at powder loading of 63.5 v/o exhibits the lowest shear rate and a very high viscosity. Meanwhile, the same feedstock at a powder loading of 64 v/o is unable to flow through the capillary rheometer die, thus no viscosity and shear rate is recorded at this particular powder loading. On the other hand, when the powder loading was reduced from 63 v/o to 62 v/o, the viscosities of melt decreased while the shear rate increased. However, Fig. 2 shows that, the shear rate of the feedstock at powder loading of 62 v/o reached a peak (8×10^3 to 9×10^3 s^{-1}) while the viscosity sank to a trough (less than 100 Pas).

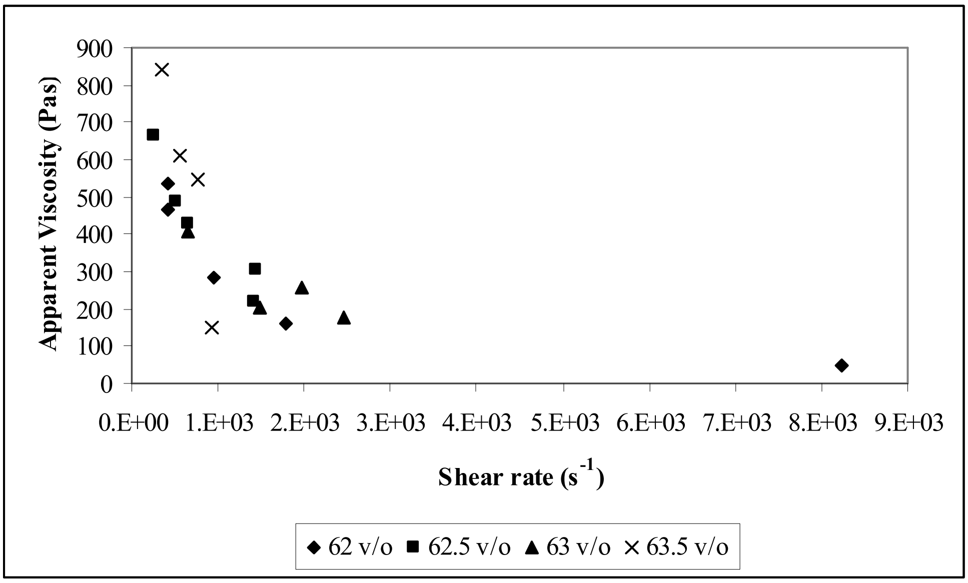


Fig. 2 Apparent viscosity and shear rate of the water atomized fine powder feedstock

Conversely, the apparent viscosity of the coarse powder feedstock shown in Fig. 3 demonstrates lower apparent viscosity than the fine powder feedstock in Fig. 2. By comparing results shown in Fig. 2 and Fig. 3, the plot in Fig. 2 only concentrates at the left hand side of the plot, but the plot in Fig. 3 is widely distributed across a wide range of the shear rate.

Moreover, as a consequence of powder binder separation that occurred in the 63.5 v/o powder loading feedstock, the viscosity of such melt steeply declined at a shear rate of $3.64 \times 10^3 s^{-1}$. However, when the shear rate goes beyond 4×10^3 s^{-1}, it can be observed that there is a slight increase of the viscosity. In addition, the coarse powder feedstock at powder loading of 63 and 63.5 v/o shows stronger viscosity and shear rate effect as compared to the same feedstock at 64 v/o. Furthermore in Fig. 3, the 64 v/o feedstock demonstrates lower viscosity than other feedstocks at a shear rate below of 4×10^3 s^{-1}. This is a consequential effect of more binder extruding through the capillary rheometer at a low shear level rate. This occurs because the ligament shaped powder particles are firmly held in the binder even at high powder loadings.

In addition, the apparent viscosity and shear rate shown in Fig. 2 and Fig. 3 indicate the feedstock's pseudoplastivity, seeing that the viscosity reduces while shear rate increases. This behavior indicates that shear thinning occurs on the feedstock, when shear stress is applied on it. The pseudoplastivity of the feedstock is shown by the flow behavior index in Table 2. Generally, if the index is smaller than unity, the melt is considered a pseudoplastic, while if it is in the reverse state, the melt dilatants.

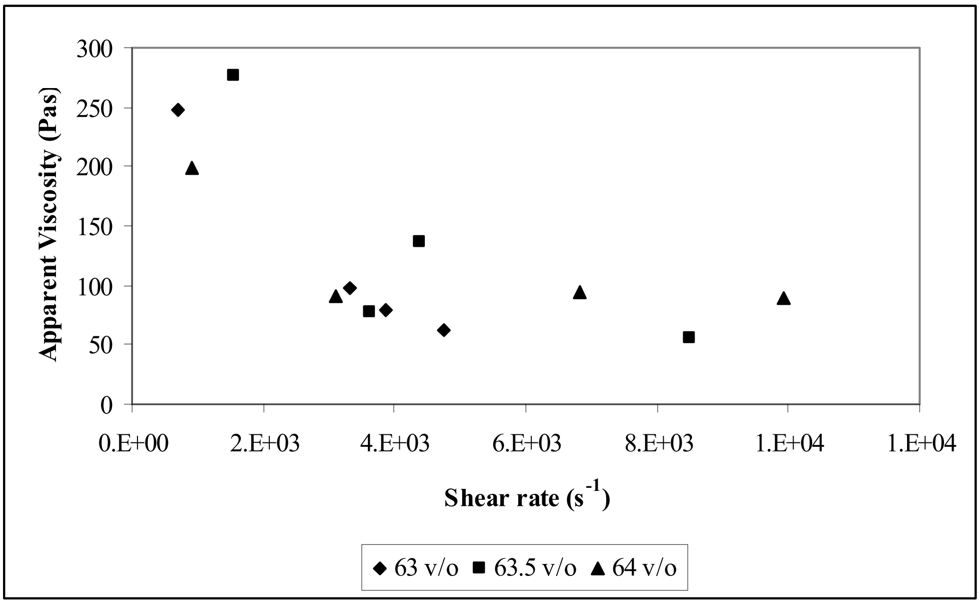


Fig. 3 Apparent viscosity and shear rate of the water atomized coarse powder feedstock

Table 2 shows the flow behavior index of the feedstock. If the flow behavior index is smaller than the unity, it indicates the pseudoplastivity of the feedstock and also the sensitivity of the feedstock to shear stress. The flow behavior index of the fine powder feedstock as shown in Table 2 displays the sensitivity of the melt at a powder loading of 62v/o will gradually increase the shear stress as the injection temperature is increasing. However, the sensitivity is found to reduce when the powder loading of the same feedstock is increased to 62.5v/o. This is probably due to the result of the dilatants flow at a high temperature. Subsequently, the same powder feedstock at powder loading of 63v/o and 63.5v/o is unable to flow through the capillary rheometer at a temperature below 130 ^{o}C and 140 ^{o}C respectively. Nevertheless, results show that at 63 v/o the shear sensitivity of the melt is reduced once the temperature is stepped up from 140 to 150 ^{o}C but, its sensitivity recovers at 160 ^{o}C.

The same phenomenon is also discovered at the powder loading of 63.5 v/o, when the temperature was raised from 150 to 160 ^{o}C. This happened probably due to small amount of PMMA which facilitates to minimize the melt viscosity burning off at 160 ^{o}C as a burning smell was discovered by the authors during the experiment. (Note that the decomposition temperature of PMMA is ≈ 170 ^{o}C.) At the subsequent powder loading for the fine powder feedstock, the test was unsuccessful even at 160 ^{o}C.

The test for the coarse powder feedstock begins from 63v/o (Table 2). Despite a small reduction of the shear sensitivity it has been indicated that at 63 v/o (140 ^{o}C), the flow behavior index demonstrates an enhancement on the shear sensitivity especially at powder loadings of 63 v/o and 63.5 v/o. Furthermore, as soon as the powder loading increases to 64v/o, a reversed trend is shown in Table 2. The shear sensitivity at powder loading 64 v/o plummeted slightly from 130 to 150 ^{o}C (flow behavior index shows slight increase) and, the cause that leads to this phenomena is not investigated in this study.

Besides, shear sensitivity being signified by the flow behavior index as shown in Table 2, activation energy is another parameter which has its significance to evaluate the moldability of the MIM feedstock. This parameter determines the feedstock sensitivity to thermal fluctuation between injector nozzles to the mold cavity. If the temperature sensitivity is too extreme, this may lead to premature melt freezing before it reaches the end point of the mold cavity. Thus, too much temperature sensitivity may deny the possibility of this process in the manufacturing of larger parts. However, this situation can be resolved if the temperature gradient from the injector nozzle to the mold cavity is improved by minimizing the thermal reduction. This can be made possible, if the mold heating mechanism on the sprue part is specially enhanced. By doing this, it can delay the melt solidification time, thereby helping the melt to travel smoothly without prematurely freezing even in bigger mold cavity. Besides that, the mold sprue line should be shorter so that thermal loss from the melt can be minimized.

Table 2 Flow Behavior Index, n water atomized powder

Powder loading (v/o)	Temp	Fine	Coarse
62	130	0.3235	
	140	0.2641	
	150	0.1988	
62.5	130	0.1241	
	140	0.3566	
	150	0.4368	
63	130	Unable to	0.6757
	140	perform test	0.8368
	150	0.3031	0.3222
	160	0.4512	
		0.3503	
63.5	130	Unable to	0.4537
	140	perform test at	0.3435
	150	temp below 140	0.0941
	160	degree	
		0.3885	
		0.0483	
64	130		0.5466
	140	Nil	0.596
	150	(unable to	0.6732
	160	perform	0.309
		rheology test at	
		this powder	
		loading)	

Table 3 shows the activation energy of the feedstock. The results indicate that the fine powder feedstock demonstrates a significant increase of the activation energy as soon as the powder loading increases from 62 to 63 v/o; although there was little reduction at 62.5 v/o possibly due to binder separation. Meanwhile, the coarse powder feedstock also shows an increase of the activation energy from 63 to 64 v/o and even the powder loading at 63.5 v/o sank significantly.

In general, coarse powder feedstock demonstrates better thermal sensitivity than the fine powder feedstock. This shows that the coarse powder has better moldability than the fine powder. Besides that, the flow behavior index as shown in Table 2 also indicates that the coarse powder feedstock is appropriate for injection molding at powder loadings of 63 and 64 v/o, especially at temperatures ranging from 130 to 150 ^{o}C. Although Table 2 shows significant flow behavior index at 160 ^{o}C, the index swiftly declines when the temperature rises to 160 ^{o}C (64 v/o). When this happens, the melt viscosity will gently increase due to the small amount of binders that are burnt out at 160 ^{o}C.

Table 3 Activation Energy, E (kJ/mole)

Powder loading (v/o)	Fine	Coarse
62	1.224	
62.5	0.952	
63	3.8296	21.51
63.5		15.36
64		28.30

4. CONCLUSIONS

The rheological behavior of the water atomized SS316L powder feedstock has been investigated. Both fine and coarse powder feedstocks exhibit pseudoplastic behavior as the viscosity decreases gradually while the shear rate increases. However, the investigation proves that the fine powder feedstock at powder loading 64 v/o was unable to flow through the capillary rheometer due to extreme melt viscosity, even when the temperature rose to 160 ^{o}C. On the other hand, the fine powder feedstock at the powder loading of 62 v/o demonstrates good pseudoplastic behavior than the one at a higher powder loading.

Alternatively, coarse powder feedstocks demonstrate lower apparent viscosity than the fine powder feedstocks. This is because more binders are attached to the ligament shape of the coarse powder particles; hence it helps the melt to flow smoothly into the mold cavity. Consequently, the coarse powder feedstock at powder loadings of 63 and 63.5 v/o show better rheological behavior compared to 64 v/o.

In addition, the flow behavior index of the fine powder feedstock at 62 v/o indicates that the shear sensitivity of the fine powder feedstock increases when the injection temperature is raised. This is a good indicator that the feedstock at 62 v/o powder loading has a better rheological behavior. On the other hand, the flow behavior index of the coarse powder feedstock demonstrates that the powder loading of 63 and 63.5 v/o is suitable to be injection molded. However, activation energy of the fine powder feedstock is extremely low compared to the coarse powder feedstock. This indicates that the temperature sensitivity of the fine powder feedstock is excessively low and therefore; there will be a possibility that a longer cooling time is required before ejecting the green part from the mold cavity. Besides that, there are other possibilities for defects that may occur on the green parts produced. These defects may vary from broken parts, to distortions and penetration of the ejector pin into the green part during ejection.

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