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Optimizing Rheological Behavior of Steel Feedstocks in Advanced Process of Alloying Powder Injection Molding

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Abstract: The rheological behavior of feedstocks used in powder injection molding technology influences strongly on the final properties of the products. Powder loading is one of the important factors that have a great distribution on rheological behaviors. By using the gas atomized spherical 316L stainless steel powder and the binder of 55% paraffin wax+ 40% polyethylen+ 5% stearic acid, four kinds of feedstocks were prepared at the powder loading of 60, 64, 68 and 72%. To find the proper feedstock and molding temperature, the applied shear rate on the samples has been increased from 100 to 1000 l/s in three different temperatures. The investigations demonstrate that 68% powder loading is the optimal one. This sample shows the minimum sensitivity to shear rate and temperature and in a wide range of temperature has a nearly steady viscosity, consequently demonstrate the best behavior during injection molding.

Keywords: Metal Injection Molding, Powder Loading, Feedstock, Rheological Behavior, Viscosity

1. Introduction

Metal injection molding (MIM) developed very fast as a kind of powder metallurgy net-shaping process in recent years [1]. The powder injection molding (PIM) typically consists of four steps. Initially, metal or ceramic powders are mixed with suitable organic binders. A binder is added between metal powder particles as the flow-vehicle to get molded parts of the desired shape. The mixture of powder and binder is termed the feedstock. In the feedstock mixture, it is expected that each powder particle, which should be enveloped by a very thin film of binder, has a tight contact with each other. At the same time, all pores among powder particles are filled with binder. But it is very difficult to get that ideal powder binder mixture. The volume ratio of solid powder to the total volume of powder and the binder is defined as the powder loading. A large excess of the binder is unacceptable because the excess binder separates from the powder during molding, leading to flashing or heterogeniety in molded parts. Most importantly, a large binder excess leads to compact slumping during debinding, since the particles are not held in place as the binder is removed. Higher powder loading means smaller compact volume shrinkage and easier dimension tolerance control, which is very important for the mass production of complex and delicate MIM parts. But too high powder loading is also unacceptable because it will lead to too high feedstock viscosity and result in the failure of injection molding [1]. In powder injection molding, higher powder volume loading is beneficial to decrease the shrinkage value in the first stage, and to get the required properties of the component in the second stage. The powder volume loading cannot be increased over a limited value [2].

Binder formulation and debinding are two most important keys for successful MIM. The use of inappropriate binders and/or debinding systems would produce defects or result in excessive processing time notably the debinding time. Binders can be classified into three categories: wax/oil-based, water-based and solid polymer solutions [3].

During molding, the feedstock flows into and fills a mold under heat and pressure to form a green part with the desired shape. The molded part then undergoes a debinding step where the polymer is

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extracted out and the powder is sintered to get full or near full density.

To fabricate specimens without cracks and distortion, the study of the rheological behavior of feedstock is very important and it is clearly influenced by powder characteristics and binder properties. The knowledge of characteristics of powder and binder is essential for successful PIM manufacturing [1].

In recent years, some investigations have been done on the influences of factors such as powder loading and temperature of the mold on rheological properties of feedstock and quality of MIM parts. Yimin Li et al. [1] studied the effect of powder loading on rheological behavior of feedstock and optimized the best one for production of proper stainless steel 17-4PH parts. Sotomayor et al. [4] studied the effect of size distribution of powder particles on the rheological behavior of feedstock and concluded that the smaller particles size and size distribution of powder particle, the more pseudo-plastic of rheological behavior of feedstock. German et al. [5] investigated the effect of powder loading on dimensional changes of MIM parts during solvent debinding step. De Souza et al.[6] got a higher powder loading with deagglomerated tungsten powder prepared by rod-milled as-received agglomerated tungsten powder. There are several papers about injection molding of stainless steels but the influence of molding temperature and powder loading on the rheological behavior of feedstock has not been deeply investigated.

The purpose of this study is to investigate the effect of powder loading of 316L stainless steel on rheological behavior of feedstock to achieve healthy and good quality MIM parts. Finding the proper temperature of molding process is another factor that has been under the study. Molding temperature affected rheological properties of feedstock and consequently has a main role in fullfilling the cavity of mold, specially at sharp edges.

2. Experimental materials and equipment

The gas atomized 316L stainless steel powder, used in the present study, was obtained from Epson Atmix Corporation. The spherical morphology of them is shown in Fig. 1. The mean particle size is 5μm. Three components binder, which has been used in the feedstock, are based on paraffin wax (PW), polyethylene (PE) and stearic acid (SA) which are produced by Kimia Exir Chemical Company. The volume ratios and physical properties of each one are shown in Table 1. PW act as filler and was used to fill the gaps among the powder particles, so it reduces the viscosity of the feedstock and facilitates injection molding. Stearic acid is a kind of surfactant which strengthens the adhesion between binder and powder, weakening the agglomeration of the powder. PE operates as backbone polymer and provides the essential strength of the green parts [7].

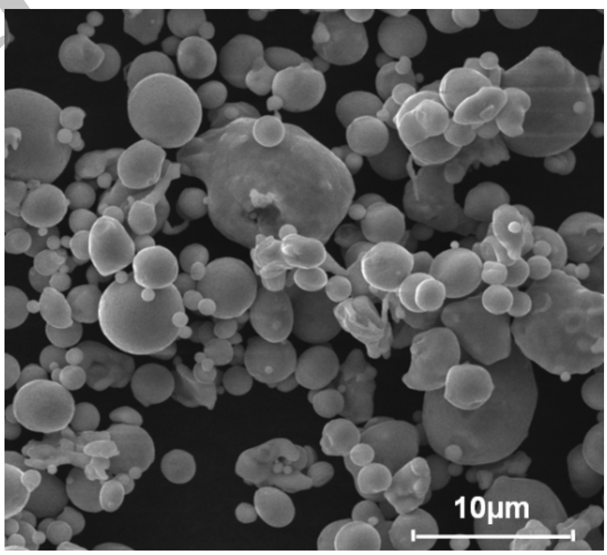

Fig. 1. Scanning electron micrograph of 316L stainless steel gas atomized powder.

To find the effect of powder loading on rheological behavior, four types of feedstocks with powder loading of 60, 64, 68 and 72 have been produced. For this case, firstly the metallic powders have been placed in the batch furnace for 1h and at 120 ºC to evaporate its moisture. Existence of moisture in the powder prevents production of homogenous feedstock for injection.

The mixtures of polymeric binder has been mixed for 10min at 140 ºC with Brabender Plastograph model mixer, then afterward the metallic powder has been added to them and has been mixed for 30 minutes at the same temperature and speed of 50 rpm. Eventually, the mixture has been used as a feedstock for injection molding. Viscosity and rheological behavior of feedstock have been investigated by capillary rheometer of MCR 300 model and made by Anton Paar factory of Austria.

3. Results and discussions

Finding rheological behaviors of feedstocks is essential to determine the capability for injection molding. The evaluation of the feedstock rheological properties is based on the viscosity and its shear sensitivity and temperature sensitivity [1]. Viscosity variation with shear rate for four feedstocks prepared with different powder loading at 130 °C are shown in Fig. 2. It can be observed that the viscosity of all the feedstocks decreases as shear rate increases, according to pseudo-plastic behavior. According to Yang's investigations [8], the decrease in viscosity with increasing shear rate causes particle (or binder molecule) orientation and ordering with flow, and may reflect improved homogeneity. Moreover, it is obvious that the viscosity of feedstocks increases by the increase in powder loading. Fluidity of feedstocks is due to the existence of binder. Therefore when the powder loading increases, the volume of binder in feedstock decreases and finally the viscosity increases. Metal injection molding feedstock showing quasi-plastic or pseudo-plastic flow characteristics have been found to be successful in MIM practice. For such fluids the relationship as Eq. (1) is valid [8]:

$$\tau = k\dot{\gamma}^{n} \tag{1}$$

Where τ is the shear stress, γ indicates shear rate, n the flow behavior index and K is a constant. $n > 1$ specifies a dilatants material where the metal powder and binder would separate under high shear rate. The most important rheological property for a metal powder feedstock is viscosity (η), which is defined by Eq. (2) as below [8]:

$$\eta = \frac{\tau}{\dot{\gamma}} \tag{2}$$

In general, the viscosity (η) is the function of shear rate (γ), temperature, powder loading and the binder compositions as demonstrated by Eq. (3) [9],

$$\eta = \eta(\dot{\gamma}, T, \Phi, \eta_b) \tag{3}$$

Where η_b is the viscosity of the binder mixture. The general shear rate-dependence of viscosity could be described by Eq. (4),

$$\eta = K\dot{\gamma}^{n-1} \tag{4}$$

Where η is the feedstock viscosity and K a constant. The slope of the log η-log γ graph is n-1, from which n can be calculated [10]. For all three feedstocks, the log η-log $\dot{\gamma}$ graphs derived from the rheological testing results are shown in Fig. 3. However the n value will be smaller, the feedstock becomes less sensitive to shear rate. Consequently, feedstock with minimum amount of n experiences the minimum changes of viscosity by variation of shear rate during injection molding. The equations of lines which are drawn in Fig. 3 are defined as Eqs. (5-8):

$$log\eta_{60} = -0.179\, log\dot{\gamma}_{60} + 3.311, \quad n_{60} = 0.821 \tag{5}$$

$$log\eta_{64} = -0.188\, log\dot{\gamma}_{64} + 3.406, \quad n_{64} = 0.812 \tag{6}$$

$$log\eta_{68} = -0.197\, log\dot{\gamma}_{68} + 3.632, \quad n_{68} = 0.803 \tag{7}$$

$$log\eta_{72} = -0.185\, log\gamma_{72} + 3.764, \quad n_{72} = 0.815 \tag{8}$$

Table 1. Volume ratios of binder components

Component	Volume Fraction (%)	Melting Temperature (ºC)	Density (g/cm^3)
Parrafin Wax	55	67	0.90
Polyethylene	40	126	0.92
Stearic Acid	5	54	0.91

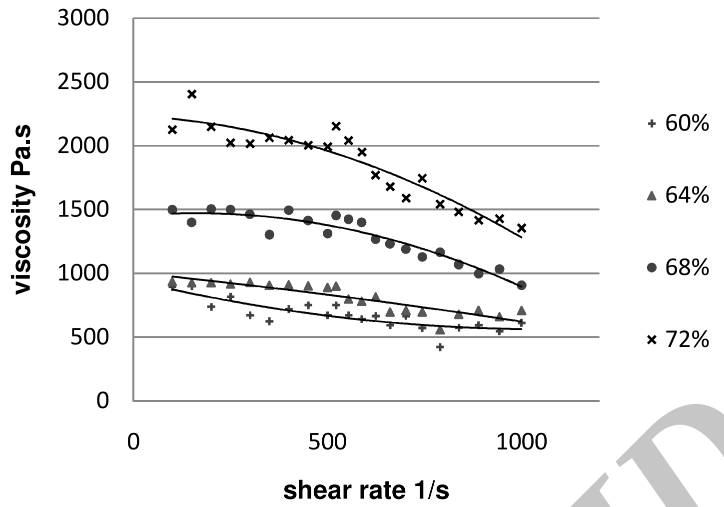

Fig. 2. Viscosity as a function of shear rate for feedstock of different powder loading at 130 ºC.

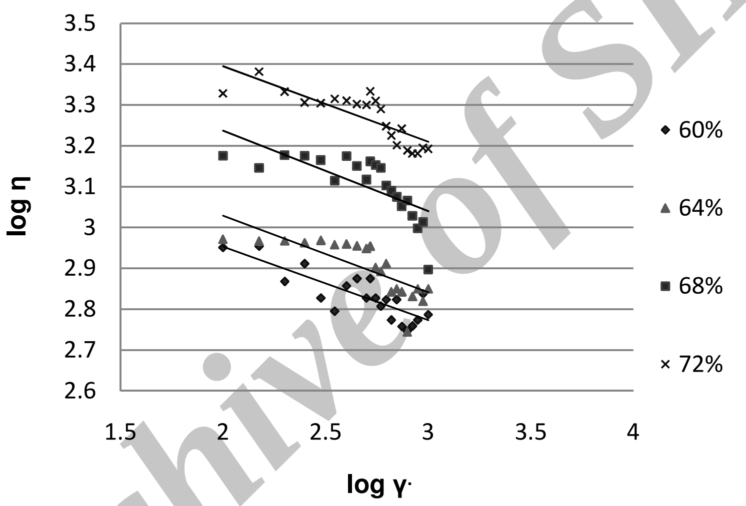

Fig. 3. Viscosity versus shear rate for feedstock of different powder loading at 130 ºC.

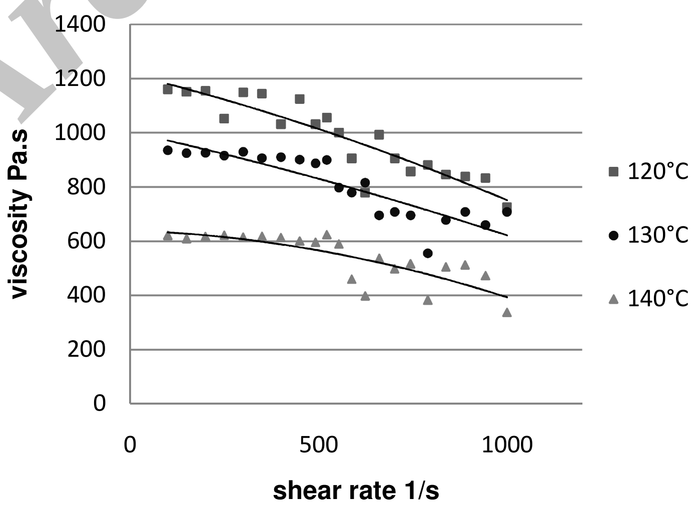

Fig. 4. Correlation of viscosity and temperature for feedstock with powder loading of 68% and different temperatures.

At first, the flow exponent of the feedstock decreases with the increase of the powder loading from 60 to 68%, achieving the lowest value of 0.803 for the feedstock with 68% powder loading. Then, the flow exponent increases with the powder loading being increased to 72%. Therefore, the lowest flow exponent for 68% powder loading indicates that there is a best powder-binder ratio for MIM feedstock for injection molding. As a general rule, there is a certain range of operating condition for MIM process. With increasing the shear rate, the viscosity decreases, which can help to reduce pressure and temperature needed for molding. In the MIM process, the shear rate during molding are usually between 100-10000 1/s. Experimental studies show that in this range of shear rates, the maximum viscosity of the feedstock at molding temperature is 1000 Pa.s [11]. Fig. 4 shows the plot of viscosity versus shear rate for feedstock of 68% powder loading at different temperatures. According to this figure, the viscosity of feedstock is permanently below the 1000 Pa.s in the common range of shear rate and at 130 ºC, consequently the temperature of 130 ºC is selected for molding.

4. Conclusions

Feedstock for proper injection molding has to contain an optimized amount of powder molding to produce an appropriate part without crack and distortion. Hence, in this study, the rheological behavior of feedstocks with powder loading of 60, 64, 68 and 72% of 316L stainless steel have been investigated.

Increasing shear rate causes reduction of viscosity and increasing powder loading causes the increase of viscosity. Plotting the changes of viscosity versus shear rate for feedstock with different powder loading shows that lowest slope or the smallest value of n is for the line of feedstock with 68% powder loading. Therefore, this feedstock has the lowest sensitivity to shear rate and have been selected for injection molding.

Temperature is another effective factor for injection molding. Researches show that the feedstock is optimized permanently in allowed range of viscosity for molding at 130 ºC. Consequently, the temperature of 130 ºC is selected as temperature of injection molding.

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