Characterization of 17-4 PH stainless steel metal injection molding feedstock using mixing torque data

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Abstract. Currently, there are many components produced by a metal injection molding process in automotive, consumer goods, medical, and electronics. Metal Injection Molding process (MIM process) consists of four stages, mixing, injection molding, debinding, and sintering. Feedstock plays critical roles in the MIM process since the feedstock’s low quality cannot be corrected later. Feedstocks, which are a mixture of powder and binder, are mixed at an elevated temperature. A feedstock should be homogeneous and has a pseudo-plastic behavior. In the MIM process, the shear rate during injection molding is usually 10 to 10,000 s\(^{-1}\). Within the shear rates range, a maximum viscosity for injection molding was 100 Pas at molding temperatures. In this paper, the rheological characteristic of feedstocks was analyzed using the torque rheometer. The objective of this research was to find the value of viscosity and compare to the Material Safety Data Sheet (MSDS) of the commercial feedstock by using torque mixing data. All the three feedstocks had pseudo-plastic behavior and below 100 Pas within shear rates range. Form the validation of injection molding experiment, feedstock B with solid loading 60 %, and binder system consists of 35 % PP, 64% PW, and 1 % SA showed a good flowability and moldability.

1. Introduction

Metal injection molding (MIM) is a manufacturing technology that uses the shaping advantage of the injection molding process to be applied to metal. The MIM products have characteristics of geometrical complexity combined with high properties because of their near theoretical density and homogenous microstructures [1-2]. MIM process is suitable for most common engineering metals such as carbon steel, stainless steel, tungsten, nickel-based alloys, titanium alloys.

The MIM process consists of four stages, mixing, injection molding, debinding, and sintering. Initially, powder and thermoplastics binder are mixed, kneaded, and pelletized to form the feedstock. Like in the plastic injection molding process, this feedstock is injected into a mold using an injection molding machine. The injection part is called the green part. The binder inside the green part is
removed by heating, chemical extraction, or catalytic reaction. The binder removal process is called as debinding process. The final process is the sintering process to produce the last densified parts.

Binders play an essential role in the final quality of MIM products. There are some binder requirements for the MIM process. The binder must wet the powder particles, melt at low temperature for the injection molding process, easily be removed without leaving residues, be decomposed at a temperature that low enough to reduce chemical reaction with metal powder, provide sufficient green strength, provide lubricity, and be environmentally friendly [2]. The binder system consists of more than one component to prevent defects during the binder removal time. The binder system includes low molecular weight, high molecular weight polymer, and surfactant. The multi-components binder allows the binder removed gradually during the debinding process without promoting the defects [3-5]. The surfactant has the function to improve the fluidity of powder and the viscosity of the feedstock. Feedstock plays critical roles in the MIM process since the quality of the feedstock affects the next process.

The MIM feedstock has a pseudo-plastic behavior that the viscosity decreases with increasing shear rate. In the MIM process, the shear rate during injection molding is usually in the range of 10 to 1000 s⁻¹. Within the shear rates range, a maximum viscosity for injection molding is 100 Pas at molding temperatures [2]. Torque rheometers (TR) are widely applied in polymers and the design of compound. The processing conditions are examined through measured torque and temperature. If the process conditions are the same, different materials flow behavior can be compared relatively [6]. The research objectives are to examine the MSDS rheology data with experimental torque data of commercial feedstock and characterize the torque and rheology properties of the feedstock with the variation of feedstock compositions.

2. Materials and methods

2.1 Materials

In this study, the 17-4 PH stainless steel (17-4 PH SS) powders were mixed with a multi-component binder consisted of paraffin wax (PW), high-density polyethylene (HDPE), polypropylene (PP), and stearic acid (SA). Stearic acid was added to act as a surfactant to the feedstock to improve the powder’s wetting. The torque rheology characteristic was compared to the commercial 17-4 PH SS feedstock. Table 1 shows the powder properties of the used powder and industrial feedstock using the Material Safety Data Sheet (MSDS). Table 2 shows the chemical composition of the metal powders using the MSDS data. The morphology of the powder can be seen in figure 1. The morphology of 17-4 PH SS powders was irregular shapes with particle size D₅₀ around 7 μm.

| Powder Particle Size | Commercial feedstock powder | Used powder |
|----------------------|-----------------------------|-------------|
| D₅₀                  | 2.8 μm                      | 2.89 μm     |
| D₅₀                  | 6.9 μm                      | 6.9 μm      |
| D₅₀                  | 18.6 μm                     | 17.91 μm    |

| Material             | 17-4PH                      | 17-4PH      |
|----------------------|-----------------------------|-------------|
| Supplier             | Epson ATMIX                 |             |

| Elements | C      | Cr     | Co     | Cu     | Fe     | Mn     | Si     | Nb     | Ni     | O      | P      |
|----------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| % Weight | 0.04   | 0.03   | 3.96   | 74.26  | 0.16   | 0.39   | 0.29   | 4.04   | 0.35   | 0.02   |
2.2 Torque rheometers

Torque caused by the resistance of a material to the applied shear is measured by torque rheometer. It is usually used for quality control and compared the rheological characteristics of the polymer and food industries. In this study, the mixing process was carried out using Rheomix from Polylab to prepare the MIM feedstock at different solid loading and binder composition. The mixing temperature was set at 190°C for 1 hour. The feedstocks were tested at different rotation speeds at specific temperatures to obtain the mixing torque data. The compositions of the mixed feedstocks are shown in Table 3. Figure 2 shows the Rheomix Polylab that used in this study and the specifications.

Table 3. Feedstock compositions.

| Feedstock   | Solid loading | Binder system |
|-------------|---------------|---------------|
|             | PW (wt %)     | PP (wt %)     | HDPE (wt %) | Stearic Acid (wt %) |
| Feedstock A | 60            | 64            | 0           | 35           | 1           |
| Feedstock B | 60            | 64            | 35          | 0            | 1           |
| Feedstock C | 55            | 64            | 35          | 0            | 1           |

- **Item**
  - Chamber Volume: 625 cm³
  - With Rotor: 310 – 541 cm³
  - Gear ratio: 3:2 (optional 2:3)
  - Max. Speed: 250 min⁻¹
  - Max. Torque: 300 Nm
  - Max. Temperature: 400°C
  - Temperature Control: 3 zones electric heating and air cooling

Figure 1. The SEM images of 17-4 PH stainless steel powder.

Figure 2. Rheomix polylab.
2.3 Shear rate and feedstock viscosity

In this paper, the commercial feedstock’s viscosity and three compositions feedstock viscosity are characterized using torque mixing data, according to Bousmina et.al. [7]. To find the shear rate - viscosity using the simple analogy of Couette for each cylinder and then calculating the overall torque given by the two cylinders in the mixed liquid. The effect of the aperture that exists between the two sets of cylinders is negligible. Then the shear rate and feedstock viscosity equations as following Bousmina et.al. [7]:

\[
\dot{\gamma} = 16\pi N \frac{\beta^2}{(1+\beta)^2(\beta^2-1)} \approx 2\pi N / \ln (\beta) \quad (1)
\]

where \(\dot{\gamma}\) is shear Rate (s\(^{-1}\)), \(N\) is rotor rotation (rpm), where \(\beta\) is the ratio of the mixing chamber \((R_c)\)diameter with rotor diameter \((R_b)\).

\[
\eta = \frac{T}{N \pi^2 L R_b^2 (1+g^2)} \quad (2)
\]

where \(\eta\) is viscosity (Pa.s), \(T\) is mixing torque (N.m), \(L\) is rotor length (m), \(g\) is gear ratio \(N_2/N_1\).

3. Results and discussion

3.1 Rheology characterization of commercial feedstock

The mixing torque is an indicator of the viscosity of the mixture. Figure 3 shows the mixing torque as a function of the mixing time at various rotation speeds at a specific temperature. Figure 3a shows the mixing torque at 170°C and figure 3b at 190°C. In the beginning, when the powder was added to the mixture, the mixing torque increased to a high value because it had not reached a steady state yet. When the mixture became homogenous, the torque reached equilibrium value. Figures 3a and 3b show the effect of the temperature into mixing torque. As the temperature increased, the mixing torque decreased.

Figure 4 shows commercial feedstock’s viscosity derived from torque data using the Bousmina model [7]. It can be seen from figure 4 that the viscosity of the feedstock declined with increasing the shear rate. The results showed that the feedstock mixture had pseudo-plastic properties following the characteristics of the MIM feedstock [8]. The viscosity of the commercial feedstock decreased with increasing temperature. Figure 4 also compared the commercial feedstock’s viscosity from the MSDS data and from mixing torque data. The results showed the same trendline between the MSDS data and calculated viscosity. However, there was a small gap between the MSDS data’s viscosity and calculated from mixing torque data. This result agreed with other researchers that the Bousmina model was best suited for pure polymers [9].

3.2 Rheology characterization of the mixed feedstock

The effects of solid loading and binder composition on the mixing torque and viscosity were examined. Figure 5 shows the mixing torque data versus rotation speed for different compositions. Figure 6 shows the viscosity calculated from mixing torque data for three different feedstocks. The results showed a similar trend for three feedstock compositions, and feedstock A and B had the same solid loading but different binder composition. Feedstock A used the HDPE as a backbone polymer, and Feedstock B uses PP as a backbone polymer. Feedstock A exhibits a higher viscosity than Feedstock B because of the HDPE had a higher viscosity than PP [10-11]. Feedstock C had the lowest viscosity among others because its low solid loading meant lower metal powder content in the mixture. Moreover, all the three feedstocks had pseudo-plastic behavior below 100 Pas within shear rate ranging from 10 to 10 000 s\(^{-1}\). Form the validation of injection molding experiment, feedstock B
with solid loading 60 %, and binder system consistsed of 35 % PP, 64% PW, and 1 % SA showed a good flowability and moldability.

**Figure 3.** Torque measurement results of commercial feedstock with the various rotation speed at a temperature a) 170°C and b) 190°C.
**Figure 4.** The calculated viscosity from torque data using the Bousmina Model.

**Figure 5.** The mixing torque for three different feedstock composition.
4. Conclusion
The rheological characteristic of feedstocks was analyzed using the torque rheometer. The mixing torque data was used to find the value of shear rate and viscosity. Using the Bousmina Model, the torque data from the torque rheometer could be converted into viscosity value. The viscosity calculated from the mixing data showed a similar trendline with the MSDS data’s viscosity value. However, the viscosity value was lower than the MSDS Data. The results in agreement with other researchers that the Bousmina Model was suitable for a pure polymer. The feedstocks (A, B, C) showed pseudo-plastic behavior and had viscosity below the commercial feedstock and fulfilled the requirement for the feedstock viscosity for metal injection molding. From the validation of injection molding experiment, feedstock B with solid loading 60 %, and binder system consisted of 35 % PP, 64% PW, and 1 % SA showed a good flowability and moldability.

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