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Influence Of Space Holder On Rheological Behavior Of Copper Feedstocks For Metal Injection Molding

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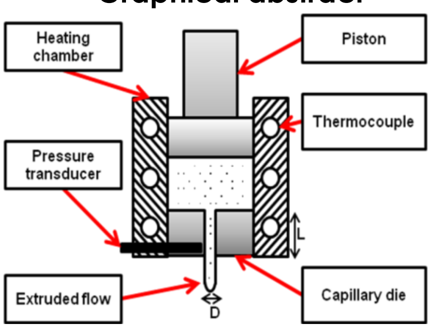
Graphical abstract



Abstract

The characterization of MIM feedstock consisting of 63 vol% of copper powder with a binder system consisting of palm stearin (PS) and low density polyethylene (LDPE) was studied. To achieve porous structure, sodium chloride (NaCl) was added as a space holder. The effect of shear rate (s^{-1}), temperature (°C) and viscosity (Pa.s) on the rheological behavior of solid and porous copper feedstocks were investigated by using the Rosand RH2000 Capillary Rheometer at temperatures of 160, 170 and 180°C. The feedstocks achieved desirable injection molding characteristics such as pseudo-plastic behaviour, stable flow, flow behavior index less than 1 (n<1) and low activation energy (E). It can be concluded from the analysis that both feedstocks showed a good pseudo-plastic behavior within acceptable ranges in MIM.

Keywords: Metal injection molding, copper, rheological behavior, viscosity, flow behavior index, activation energy.

1.0 INTRODUCTION

Metal injection molding (MIM) is a powder metallurgy forming process using metallic powder which have key features such as low production cost, shape complexity, tight tolerances, applicability to several materials and excellent final properties . There are 4 main sequential processes of MIM which are mixing, injection molding, debinding and sintering.

During molding, the shear rate at the molding temperature usually ranges between 10^2 and 10^5 s^{-1} and the maximum useful viscosities for the mixture is 1000 Pa.s or lower]. The molding stage is the critical step for fabricating 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 [.

Previous study of solid copper feedstock by Nor Amalina Nordin concluded that all the copper feedstock posses a pseudo-plastic behavior with the viscosities decreasing with increasing shear rate [. A study on rheological behavior containing copper/carbon nanotubes by Faiz Ahmad showed that although the viscosity was more than 1000 Pa.s, high injection pressure in injection molding stage managed to produce a defect free sample. Another study conducted by M. Azmiruddin on MIM of Inconel 718 using PS based binder system showed that higher concentration of PS in the binder system reduced both the activation energy and viscosity of the feedstock. Meanwhile, Iriany reported that feedstock with binder formulation containing palm oil greater than 45wt% showed instability of the feedstocks. Despite several investigation on the rheological properties of solid feedstocks such as copper, stainless steel, Inconel 718, titanium alloy in MIM, studies on influence of space holder material on rheological behavior feedstocks were rarely reported.

This study aims to evaluate the rheological properties of feedstock containing sodium chloride as space holder in terms of pseudo plastic behavior, shear sensitivity and activation energy. Therefore, this study is highly significant to obtain new knowledge and data related to rheological behavior of solid and porous copper feedstock.

2.0 EXPERIMENTAL

2.1 Materials

Gas atomized copper powder used in the present study was obtained from Sandvik Osprey Powder (Figure 1). The mean particle size D_{50} is 14.2µm and was determined using a Malven Particle Size Analyzer (Table 1). The binder system consisted of a mixture of LDPE which was purchased from Lotte Chem. Company while PS which was obtained from Sime Darby Plantation was kept constant for all feedstocks. Characteristic of binder components such as melting temperature and degradation temperature, which were obtained from DSC and TGA are shown in Table 2. To achieve porous copper part, sodium chloride was used as a space holder. Sodium chloride is being filtered to have sodium chloride powder size not exceeding 100µm (Figure 2).

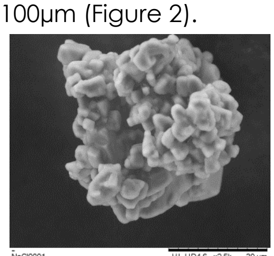


Figure 1 SEM micrograph of Copper powder

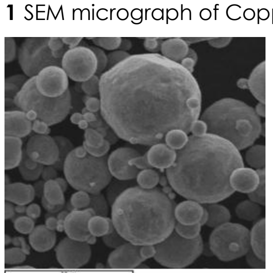


Figure 2 SEM micrograph of NaCl powder

Table 1 Results from Particle size analyzer

Copper powder	D_{10}	D_{50}	D_{90}
Size (µm)	9.047	14.2	20.94

Table 2 Characteristics of binder and space holder component.

Space Holder Component	Binder Component	Density (g/cm³)	Melting Temperature (°C)	Degradation Temperature (°C)
-	PS	0.89	59.93	382.09
-	LDPE	0.95	114.42	446.64
NaCl	-	2.17	801	-

2.2 Feedstocks Preparation

Two feedstock formulations of 63 vol% powder loading to the binder system consisting of LDPE and PS were mixed using z-blade mixer at a temperature of 170°C for 2 hours with av rotation frequency of 70 rpm to ensure homogeneity of the feedstock is achieved. The feedstock formulation without space holder and the one containing space holder were labeled as S and P respectively and tabulated in Table 3. LDPE acts as a backbone polymer which retains component's shape during the debinding process while PS provides fluidity of the feedstock during injection molding.

Table 3 Composition of feedstock (vol%)

Formulation	Powder Loading (Vol%)	Copper (Cu)	Sodium Chloride (NaCl)	Palm Stearin (PS)	Low Density Polyethylene (LDPE)
S	63	100	0	60	40
P	63	80	20	60	40

2.3 Viscosity Measurement

The test was conducted using a RH Rosand 2000 capillary rheometer. The test was conducted at temperatures of 160, 170 and 180°C with the capillary die of 1mm diameter and length of 10mm (L/D=10). The results of viscosity and shear rate were measured in Pa.s and 1/s units respectively. Figure 3 shows a schematic diagram of capillary rheometer and Figure 4 shows and parameters in data acquisition from capillary rheometer barrel in Rosand Flowmaster user interface. The example of post-extrusion condition of the feedstock as shown in Figure 5.

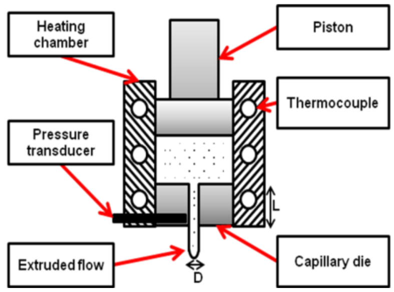


Figure 3 Schematic diagram of capillary rheometer

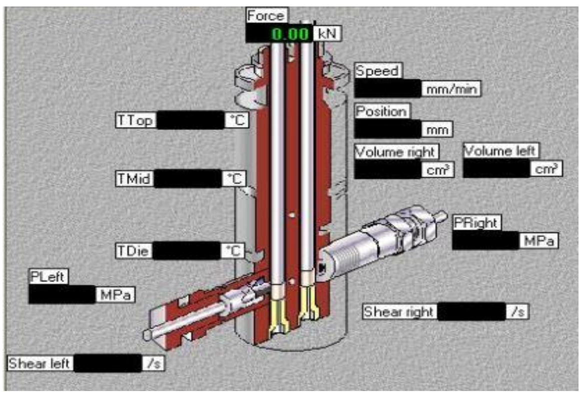


Figure 4 Parameters in data acquisition from capillary rheometer barrel into Rosand Flowmaster user interface

Figure 5 Example of post-extrusion condition of the feedstock

3.0 RESULTS AND DISCUSSION

3.1 Viscosity

The comparison of rheological properties of two different feedstocks with same powder loading of 63Vol% at temperatures of 160°C, 170°C and 180°C are presented in Figure 6. In this study, the temperatures chosen were above the melting temperature of the binder components as it is crucial to ensure that the viscosity measurement is performed at a temperature range greater than the melting point of the binder components as tabulated in Table 2. Figure 6 depicts that viscosity of both solid and porous copper feedstocks decreases with increasing shear rate. This behavior is in good agreement with pseudo-plastic behavior which satisfies one of the initial requirements of good binder system. The pseudo-plastic behavior of the feedstock suggests that the fluid binder released helps to break the particle agglomerates which in turn leads to particle re-orientation and ordering as the feedstock flows within the die. Moreover, the viscosity values obtained from both feedstocks were less than 1000 Pa.s. which is favorable in MIM practices

The trend of starting viscosity values for both solid and porous feedstocks is directly proportional to the setting temperature. For solid copper feedstock, the highest starting viscosity value was observed at a temperature of 180°C with viscosity value of 326 Pa.s followed by temperatures of 170°C and 160°C which exhibit viscosity values of 287.3 Pa.s and 265.46 Pa.s respectively. With the presence of 20 vol% space holder in the feedstock, the viscosity values increased particularly at low shear rate. Porous copper feedstock possessed the highest starting viscosity value which is also at a temperature of 180°C with viscosity of 657.67 Pa.s followed by temperatures of 170°C and 160°C which exhibit viscosity values of 552.03 Pa.s and 561.04 respectively.

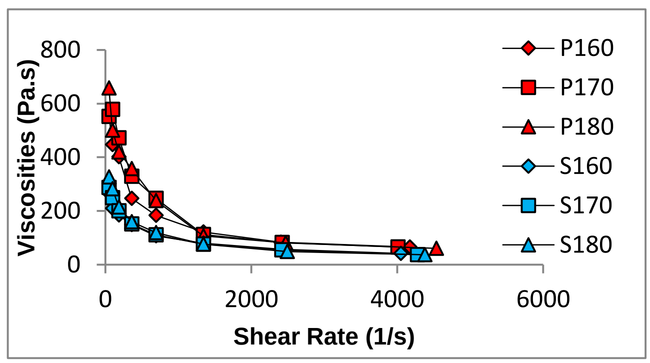


Figure 6 Viscosity versus shear rate of solid and porous copper feed stocks tested at 160, 170 and 180°C.

3.2 Flow Behavior Index (*n*)

The relationship between viscosity and temperature of the feedstock can be determined by using the following mathematical equation:

$$\eta = K\gamma^{n-1} \quad (1)$$

where η is the feedstock viscosity, γ the shear rate, K is a constant and *n* is the flow behavior index (smaller than 1). The degree of shear sensitivity of a feedstock is indicated by the value of flow behavior index, *n*. The lower the value of *n* means the more the viscous dependence on shear rate. The value of *n* should be smaller than 1 to show the presence of shear thinning behavior in pseudo-plastic materials. Meanwhile, flow behavior index greater than 1 indicates a behavior of dilatants materials which leads to separation of powder and binder under high shear rate.

The slope of the plot of log η against log γ shown in Figure 7 is *n*-1, in which *n* can be deduced. The value of flow behavior index, *n*, for all feedstock formulations depicted in Figure 8 which is lower than 1 suggested pseudo-plastic behavior.

The findings from the present study suggest that the shear rate had moderately affected the viscosity of the feedstock as the average value of flow behavior index for both solid and porous feedstocks were 0.54 and 0.46 respectively compared to flow behavior index of 0.75 found by M. Khakbiz who studied the stainless steel 316L feedstock using waxes as a major binder

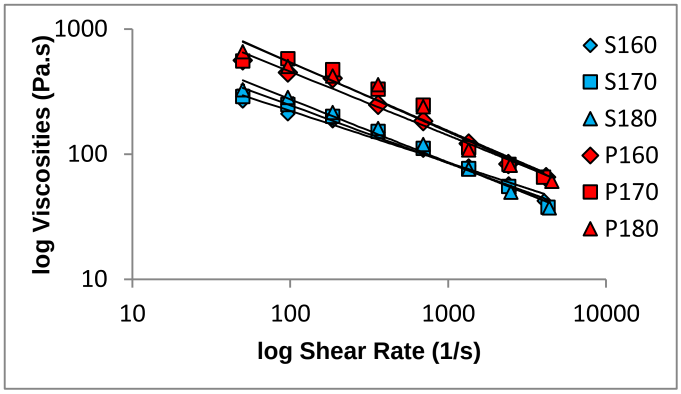


Figure 7 Log Viscosity versus Log Shear Rate for solid and porous copper feedstocks.

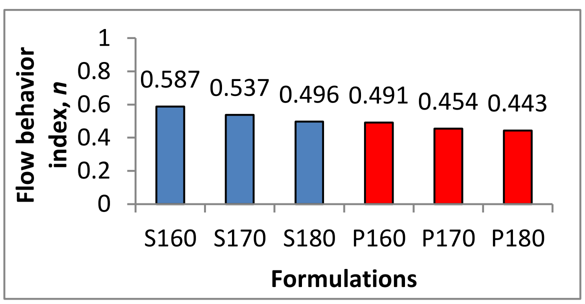


Figure 8 Flow behavior index for solid and porous copper feedstocks at temperatures of 160, 170 and 180°C.

3.3 Activation Energy (E)

Another important characteristic of MIM feedstock is temperature-dependence of viscosity. For a feedstock which is very sensitive to the temperature variation, any small fluctuation of molding temperature could lead to viscosity change which eventually may cause cracking and distortion of the injection molded parts. The influence of temperature on the viscosity of the feedstock is given by the mathematical equation:

$$\eta = K\,\gamma^{n-1}\exp(E/RT) \qquad (2)$$

where E is activation energy and R is gas constant.

Low activation energy, E, is related to low dependency of viscosity to temperature. As depicted in Figure 9, for solid feedstock it is visible that the value of E decreases as shear rate increases. Meanwhile for porous copper feedstock, the presence of space holder reduces the activation energy, which is desirable in MIM practices. The porous copper feedstock recorded the highest value of E, 22.67 kJ/mol at shear rate of 695/s while the lowest value of E, 0.068 kJ/mol was recorded at a shear rate of 2400/s. Investigation of porous NiTi alloy using binder of polyethyleneglycol (PEG) by M. Hussain Ismail showed that all feedstocks had E values in the range of 40-80 kJ/mol which can be considered high compared to the present study of solid and porous copper feedstock

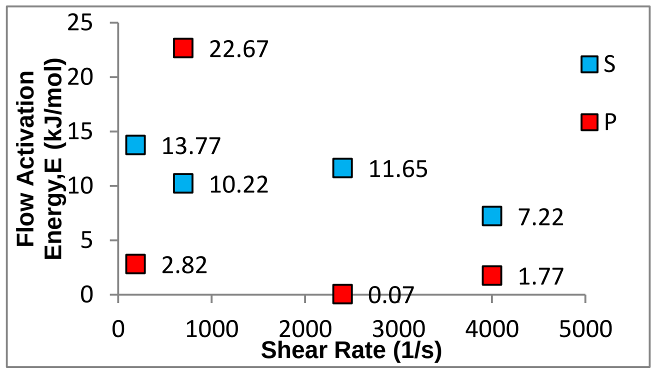


Figure 9 Flow activation energy of solid and porous copper feedstocks as a function of shear rate.

4.0 CONCLUSION

The findings from this study show that both solid and porous copper feedstocks exhibit shear thinning or pseudo-plastic behavior at all temperatures and no dilatant behavior or shear thickening was observed. The addition of sodium chloride (NaCl) as space holder increased the viscosity of the feedstock and reduced the dependency of feedstock viscosity on temperature. Hence, porous feedstock have lower activation energy (E) compared to solid feedstock which is desirable in MIM practice. Nevertheless, both solid and porous copper feedstocks are suitable for further injection molding stage.

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