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Preparation and Feedstock Characterization of Starch based Binder for Injection Moulding of 316L Stainless Steel

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Abstract – Characterization of feedstock for Metal Injection Moulding (MIM) process is essential and ensures the quality of the injection moulded parts. This study focuses to investigate the characteristics of MIM feedstock consisting of 316L stainless steel powder mix with binder system comprising of paraffin wax (PW), rice starch (RS), stearic acid (SA) and low density polyethylene (LDPE). Particle size analysis, scanning electron micrograph (SEM), thermogavimetric analysis (TGA) and differential scanning calorimeter (DSC) were performed in order to determine the characteristics of stainless steel 316L powder and binder components. The feedstock were prepared using powder loading of 0.61, 0.63, 0.65 and 0.67 with binder formulations of 50% PW-30% RS-10% SA-10% LDPE and 40% PW-40% RS-10% SA-10% LDPE. Highest mixing torque was gained with powder loading of 0.63 stainless steel 316L powder and binder system comprising of 40% PW-40% RS-10% SA-10 % LDPE. Rheological results performed pseudoplastic behavior, where its viscosity decreased with increasing shear rate. The best feedstock with good rheological characteristics is the 0.61% powder loading feedstock with binder formulation 50% PW-30% RS-10% SA-10% LDPE. The feedstock should be injected at 95°C due to its low viscosity values which is less than 1000 Pa.s. It also performed lowest value of flow behaviour index, n and higher mouldability index which preferable as feedstock for MIM.

Index Terms - Metal Injection Moulding, characterization, feedstock, stainless steel 316L.

1. INTRODUCTION

Metal injection moulding (MIM) is a process which is similar with plastic injection moulding process but differs in type of material usage. This advanced process is a modification of common plastic injection moulding in the way of MIM uses metal-based material compared to plastic injection moulding which uses plastic-based material. MIM is a process where metal powder is mixed with a measured amount of binder component to produce feedstock which then to be injected through injection moulding machine. This process allows metal-based complex parts to be produced in a single operation and end up with highly precision tolerance product. MIM is a new technology and often mistakenly compared with powder metallurgy or die casting.The MIM process has four main sequential process starting with mixing process, injection moulding process, debinding process and sintering process. The major advantages from this new manufacturing technology include high product density, more complicated shape, higher mechanical properties, and better surface finish than traditional powder metallurgy products. Moreover, an inherent advantage of MIM is that the moulding parts are hard enough to meet any needs for secondary machining [1].

However, MIM process is rarely used in order to produce parts, but it gains its popularity in recent years due to several understandings of the MIM process have been achieved. One of the most important understandings is the characterization of feedstock. Characterization of feedstock is one of the important tasks in order to evaluate the homogeneity level of the feedstock prepared and to control the quality of the parts during injection moulding process [2]. This paper covers the initial step of preparation of stainless steel 316L feedstock including characterization of metal powder, binder components and feedstock. Different percentages of volume and different binder formulations are used in this paper to study the flow ability of feedstock through rheological studies.

2. MATERIALS AND METHOD

2.1 Characterization of Metal Powder

In this study, stainless steel 316L powder by Sandvik Osprey with size range of 22µm was used. Particle size distribution analysis of the metal powder was performed by using Cilas Laser Particle Size Analyzer. Scanning Electron Microscope (SEM) was used to investigate the morphology of the powder. The average density of the powder was obtained using AccuPyc II 1340 Gas Pycnometer

2.2 Characterization of Binder Components

Characterization of binder components was essential in this research. The thermal analysis of the binder was first carried out using two common characterization techniques which are differential scanning calorimeter (DSC) and thermogavimetric analysis (TGA). DSC was conducted to determine the onset and peak melting temperature of the binders while TGA was used to determine the weight changes in relation to a temperature program in a controlled atmosphere [3].

2.3 Preparation of Stainless Steel 316L Feedstock

The metal powder and binder components were mixed using Brabender Plasti-Corder. The mixing time was 2 hours with speed 50 revolutions per minute (rpm). The mixing temperature was set to be 160°C. The binder system consists of four components which are paraffin wax (PW), rice starch (RS), stearic acid (SA) and low density polyethylene (LDPE). There were two binder formulations for each powder loading which are 50% PW-30% RS-10% SA-10% LDPE and 40% PW-40% RS-10% SA-10% LDPE. Table 1 shows the selected powder loading and the binder formulation for each feedstock.

2.4 Thermal Analysis of Feedstock

The feedstock produced then was characterized using the same two common characterization techniques which are differential scanning calorimeter (DSC) and thermogavimetric analysis (TGA). TGA is very useful to determine the degradation of binders and also to design the thermal pyrolysis cycle for debinding.

Feedstock	Powder Loading Vol %	Binder Formulation (wt %)
F1	61	PW: 40 Rice starch:40 Stearic acid:10 LDPE:10
F2	63	
F3	65	
F4	67	
F5	61	PW: 40 Rice starch:40 Stearic acid:10 LDPE:10
F6	63	
F7	65	
F8	67	

Table 1: Binder Formulations

2.5 Rheological Analysis of Feedstock

After TGA and DSC, the characterization of feedstock continued with rheological study. This was performed by using Bohlin Instruments RH2000 Capillary Rheometer. Throughout the capillary rheometer testing, the feedstock's viscosity was decreasing as the shear rate increasing. The feedstock exhibits pseudoplastic behavior if its flow behavior index was less than 1 [3].

3. RESULTS AND DISCUSSIONS

3.1 Characterization of Stainless Steel 316L Powder

Figure 1 shows the morphology of the stainless steel 316L powder from SEM at magnification of 1000X used in this study. It shows that the powder is almost in spherical shape. This shape induces to provide a maximum packing density, exhibits good mouldability, and lowers the powder/binder viscosity. Therefore it is preferred shape for MIM [4]. Furthermore, spherical shape particle is preferable for MIM since more packing is possible than with irregular shaped particles. Therefore, the preferable shape for metal powders is round and wide size-distributed [5,6,7].

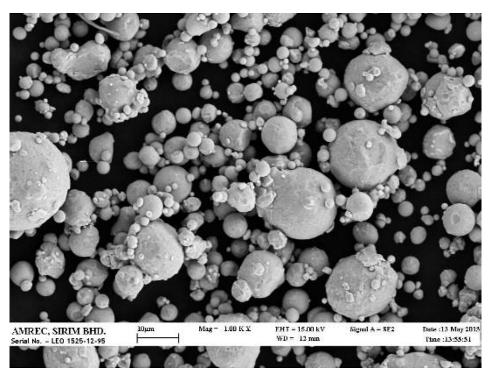


Figure 1 Scanning Electron Micrograph of stainless steel 316L powder

Wide-size distributed is a good characteristic for a powder. Thus, the particle size distribution of the metal powder was done by using Cilas Laser Particle Size Analyzer. The results from the particle size analysis were tabulated in Table 2. The data showed the particle size distribution of stainless steel 316L powder is in range between 2.39µm to 12.73µm. The average density obtained for the powder is 7.93g/cm^3.

Diameter (%)	10	50	90
Particle size diameter	2.39 µm	6.03 µm	12.73 µm

Table 2 The cumulative particle size distributions of SS 316L powder

3.2 Rheological Anlaysis of Feedstock

Typically in MIM process, shear rate varies in the range between 100 and 100000 s^{-1}. In this range, viscosity of a preferable feedstock during moulding has to be lower than 1000 Pa.s [1].

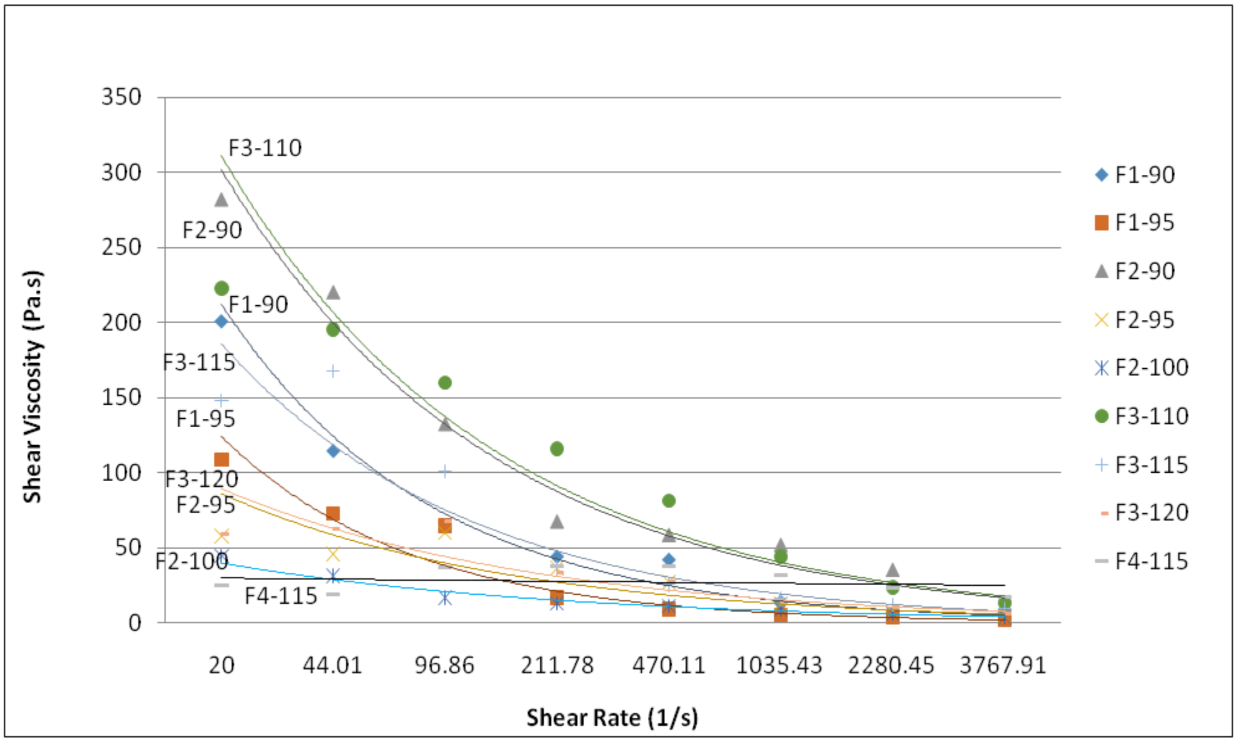


Figure 2 Graph Shear Viscosity versus Shear Rate at different temperatures

From the rheological analysis, almost all feedstocks showed pseudoplastic behaviour and the viscosity lies between 10 and 1000 Pa.s which is preferable for injection moulding process. F1, F2, F3 and F4 had been tested with a few temperatures range from 90°C to 120°C. The temperatures which cause difficulties during rheological test were eliminated. Meanwhile, F5, F6, F7 and F8 had difficulty to be injected thorugh the die. This can be explained due to lack of paraffin wax quantity which it is used to induce easy flow of metal powder into mould cavity [9]. Thus, the feedstocks were not feasible for MIM process. Figure 2 depicts the flow curves of feedstocks F1, F2, F3 and F4 at different temperatures which are in range of 90°C to 120°C.

It was found from the curves that the viscosity decreases with increasing shear rate. This shown pseudoplastic behaviour which is the most general kind in non-Newtonian fluids [1]. For non-Newtonian fluids, the relationship between shear viscosity and shear rate could be described by the Equation (2);

$$\eta = K \gamma^{n-1} \qquad (2)$$

where K is a constant, γ is viscosity and n is the flow behaviour index. The n value determines shear dependency of viscosity. Unlike Newtonian fluids, viscosity of non-Newtonian fluids varies with increase or decrease shear rate. Dilatant fluids (n>1) exhibit an increase on meeting ascended shear rate. Meanwhile, in pseudoplastic substance (n<1), the viscosity decreases with increasing shear rate [1]. The flow behaviour index, n for each feedstock obtained was then being evaluated.

Table 3: Flow behaviour index and shear viscosity for F1-F4

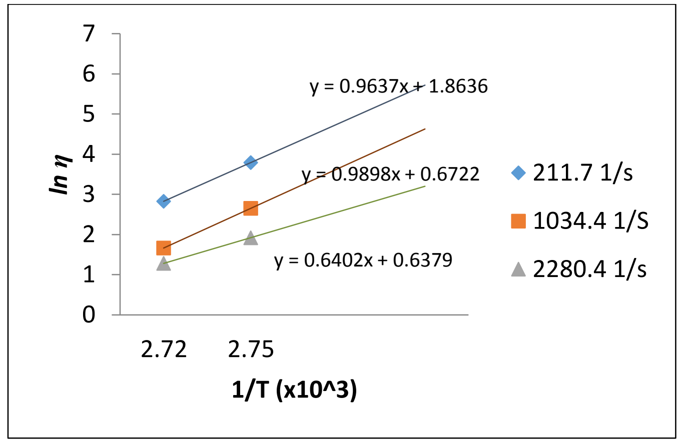
Feedstock	Temperature, °C	Shear Viscosity, Pa.s	Flow behaviour index, n
F1	90	201.18	0.58
	95	2.51	0.31
F2	90	281.63	0.61
	95	57.82	1.09
	100	4.01	0.65
F3	100	222.94	0.93
	115	9.7	0.46
	120	59.21	0.95
F4	115	25.26	1.46

Table 3 shows the flow behaviour index, *n* and shear viscosity value for F1-F4. Then, the lower *n* value for each feedstock at a specified temperature was chosen. If the flow behaviour index, *n* is greater than 1, the feedstock is not feasible as a feedstock for MIM. The feedstocks which had lower *n* value were F1-95, F2-90 and F3-115. The viscosity value for each feedstock as above mentioned was then evaluated. F2-90 has exhibit a higher viscosity than others which is not preferred for MIM feedstock. Temperature dependency of viscosity is the next significant factor which is very desirable in determination of the fluid characteristics. With a good approximation, Arrhenius equation can be utilized to describe correlation of viscosity and temperature:

$$\eta = \eta_o \exp\left(\frac{E}{RT}\right) \quad (3)$$

where η_o is viscosity at reference temperature, E is activation energy, R is the universal gas constant and T is the temperature.

Flow activation energy can be derived through correlation between viscosity and temperature. Thus, the activation energy of F1 and F3 had been evaluated. The temperature dependency of two acceptable feedstocks, F1 and F3 has been drafted at three sets of shear rates including 211.7, 1034.4 and 2280.4 s^{-1} in a graph. 211.7, 1034.4 and 2280.4 s^{-1} were selected because shear viscosity was obtained at the selected shear rates for both feedstocks. The graph "ln η versus 1/T" was plotted in Figure 3(a) and 3(b) to calculate the activation energy. The slope obtained from the graph is equal to E/R where E is the activation energy and R is the universal gas constant.



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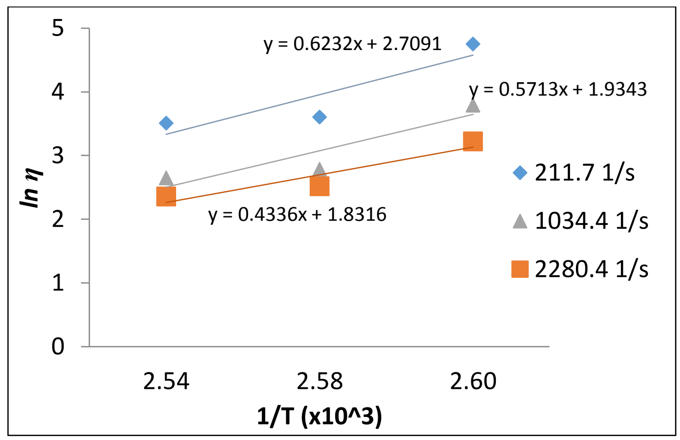

b

Figure 3: Graph "ln η vs 1/T" for (a)F1 and (b)F3

The results tabulated in Table 4 clearly shows that F1 needs higher energy to flow when compared to F3. The value of activation energy, *E* indicates the sensitivity of the viscosity to temperature. When the activation energy is low, the less sensitive viscosity to temperature variation . If the viscosity is very sensitive to the temperature variation, any small fluctuation of temperature during injection moulding results in a sudden viscosity change. This can cause defect in the moulded part, resulting in cracking and distortion. Therefore, lower *E* of feedstock is prefferable for injection moulding which means F1-95 is not preferable feedstock because it has higher flow activation energy.

Table 4: The flow activation energy of feedstock (kJ/mol)

Shear rate (s^{-1})	**F1**	**F3**
211.7	8.01	5.32
1034.4	8.23	4.75
2280.4	5.32	3.6

Meanwhile, a feedstock with low viscosity, low flow behaviour index and low activation energy exhibits better rheological properties for injection moulding. However, sometimes there is a contradiction among these parameters. Hence, another parameter has to be evaluated. The mouldability index, α_{STV} has been established to integrate the effect of all above mentioned factors. Practically, Equation (4) was used:

$$\alpha_{STV} = \frac{1}{\eta_o}\frac{|n-1|}{\frac{E}{R}} \quad (4)$$

where S is for shear, T is for temperature and V is for viscosity respectively.

It has been stated that the higher values of α_{STV} the better general rheological properties [8]. Table 5 shows the mouldability index obtained for F1 and F3 at 1034.4s^{-1} as a function of temperature. 1034.4s^{-1} was selected as reference because the shear rate has been used previously to obtain value of *E*. Thus, the value of *E* obtained at 1034.4s^{-1} was used to calculate the moudability index.

Table 5: Mouldability index for F1 and F3

Feedstock	**Temperature (°C)**	**Mouldability index, α_{STV}**
F1	90	5.90 X 10^{-2}
	95	13.99 X 10^{-2}
F3	110	2.88 X 10^{-2}
	115	6.05 X 10^{-2}
	120	7.91X 10^{-2}

The results obtained in Table 5 clearly shown that mouldability increases when increment of T. Feedstock with 0.61% powder loading (F1) shows higher mouldability index compared to feedstock with 0.65% powder loadling (F3). This means F1 is more preferable compared to F3 although high powder loading is more suitable to produce compacts with minimum dimensional shrinkage. Feedstock with 0.61% powder loading at 95°C shows the highest mouldabilty value, thus it was the most suitable feedstock to be injected at appropriate injection moudling parameter.

4. CONCLUSION

The characterization and rheological properties of feedstock consisting of 316L stainless steel powder mix with binder system comprising of paraffin wax (PW), rice starch (RS), stearic acid (SA) and low density polyethylene (LDPE) has been investigated. The mixing parameters were based on the DSC analysis of binder components. It can be seen that almost all feedstock showed pseudoplastic behaviour which is the desirable in MIM process. Among all the feedstocks, the best feedstock with good rheological characteristics is the feedstock with 0.61% powder loading and binder formulation comprising of 50% PW-30% RS-10% SA-10% LDPE. The feedstock should be injected at 95°C due to its low viscosity values which is less than 1000 Pa.s , the lowest value of flow behaviour index, n and higher mouldability index.

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