

Flow Behavior and Injectability of SS17-4PH with PS and PE Binders in Powder Injection Molding

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ABSTRACT

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Mass produced of desired molded parts is possible with Powder Injection Molding (PIM). This work focused on the rheological properties of stainless steel (SS) 17-4PH with a binder system of palm stearin (PS) and polyethylene (PE). The critical powder volume percentage of SS17-4PH at 75vol% was obtained. Based on such value, the powder loadings used in this work were 71vol%, 72vol% and 73vol%, were mixed with 60wt% PS and 40wt% PE. A capillary rheometer was employed for the rheological studies where the relationship between shear rate and viscosity was investigated. In addition, the value of flow behavior index, n for each powder loading was also computed in order to identify the injectability and activation energy, E of feedstocks. It was found that powder loading at 72vol% gave the best rheological properties.

Keywords:

Rheology, powder injection molding, stainless steel

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1. Introduction

Powder injection molding (PIM) is a powder metallurgy process that allows complex shaped parts to be mass manufactured. Such method is possible with polymers, metals, ceramics or composites [1]. Four main steps are involved in PIM such as mixing, injection molding, debinding and sintering. SS17-4PH is a magnetic and martensitic precipitation hardening stainless steel that has high strength and good corrosion resistance. It is widely used in medical, automotive and aircraft applications. However, the use of SS17-4PH in PIM is less reported. Therefore, the aim of this study is to investigate the suitability of PS and PE as binders for SS17-4PH in terms of rheological behavior and injectability in PIM for electronic applications.

Prior producing the feedstock, CPVP is conducted using the oil absorption method based on ASTM D-281-31 [2]. Fine powders usually give lower CPVP due to higher interparticle friction. Furthermore, fine powders tend to agglomerate that slows down the debinding process. In addition, injection process may not be possible if feedstock at its CPVP is used due to high viscosity [3]. Nevertheless,

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rheological properties are important to be examined prior the injection molding process which has often been characterized by capillary rheometer. The mechanical properties of the green part is also investigated. Such properties are greatly depends on the binder formulations and its compositions.

The purpose of rheological properties investigation is to ensure the mold filling of PIM feedstock into the die cavity is dependent on the viscosity of such feedstock [4]. Therefore, viscosity is a crucial factor for the feedstock to flow into the die cavity efficiently. Besides viscosity, other researchers also claimed that low shear rate indicates a good rheological behavior [5]. The preferred behavior is called pseudoplastic flow where the viscosity decreases with the increasing shear rate [4]. In addition, such flow helps to produce green parts having uniform density with less defects [6].

2. Methodology

SS17-4PH powder with the average size of 22 μm and pycnometer density of 7.78 g/cm^3 is supplied by Osprey Co, UK. The binder system consists of 60wt% PS and 40wt% PE. The function of PE is to help in decreasing the viscosity of feedstock and to increase the replication ability [7]. PE also acts as a backbone polymer which retains the component shape during the debinding process while PS provides the fluidity of feedstock during the injection molding process. CPVP of feedstock is then determined using a mixer (Brabender) which provides the optimum powder loading that suite the feedstock stability and avoid powder-binder separation.

Three feedstocks at 71vol%, 72vol% and 73vol% powder loadings are mixed with the binder system. The mixing process is also conducted using the same mixer at 150°C for 2 hours with a rotational torque of 25 Nm in order to ensure a homogeneity of the feedstock. The rheological properties of feedstock is then verified using a capillary rheometer (Shimadzu) where the viscosity, flow behavior index, activation energy and moldability index, are obtained. Low viscosity is preferred so that the feedstock can flow before it solidifies. The rheology test is conducted at three different temperatures; 130°C, 150°C and 170°C. The loads are varied from 30kgf to 90kgf.

The 72vol% feedstock is selected to be injected due to the lowest value of activation energy, E obtained from the rheology test. The feedstock is then injected into a dumbbell shape using a plunger-type injection molding machine (DSM Explore). Two stages of debinding are involved where solvent debinding is carried out first followed by thermal debinding. Solvent debinding is conducted by immersing the injected parts into heptane solution at 60°C for 4-5 hours to remove PS. PE is then removed by thermal debinding which is conducted at 500°C for 7-8 hours.

After the injection molding is successfully conducted, a three-point bending flexural test is conducted by using a Universal Testing Machine (Instron 5567). The speed and load are 0.1mm/min and 30kN, respectively.

3. Results and Discussion

3.1 Rheological Properties

Based on all feedstocks, it is found that 72vol% feedstock has the best rheological properties where such feedstock showed pseudoplastic flow as shown in Figure 1. It is observed that the viscosity decreases with the increasing shear rate. Although the preferred range of viscosity in PIM is less than 1000 Pa.s, the obtained values which are more than 1000 Pa.s, are still acceptable due to the pseudoplastic flow type. Bigger value of viscosity may due to the higher fraction powder of powder used for the feedstock.

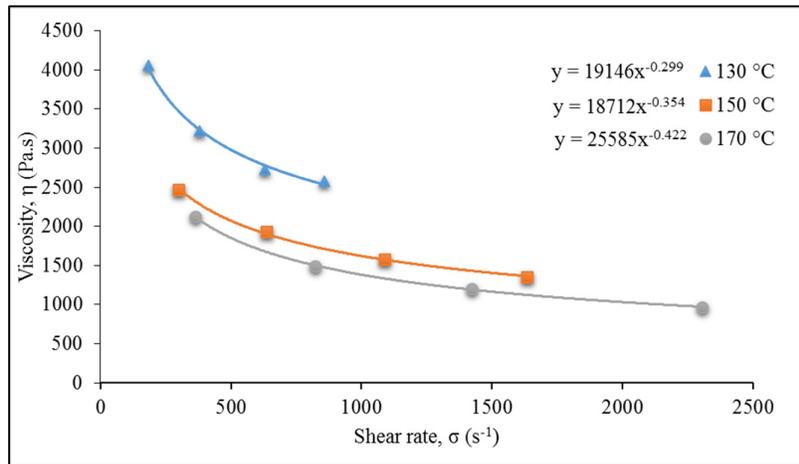


Fig. 1. Variations of viscosity and shear rate for 72vol% feedstock at three different temperatures; 130°C, 150°C and 170°C

Such flow also indicates a good agreement that the PS and PE binders are suitable for the SS17-4PH powder. Such binders also help to break the agglomerated particles powder so that a better ordering of particles can be achieved as the feedstock flows into the die cavity [9]. Besides, such flow also helps in retaining the shape of the green part, easing the mold filling and minimizing jetting [10]. In addition, the temperature and pressure during the injection molding can also be reduced.

The viscosity of feedstock decreases as the temperature increased due to the expansion of binder [11]. Too high temperature may lead to molecular disentanglement that occur due to the fluctuation of random molecular structure [10,12]. The relation of such flow is determined by the power law equation (Equation 1)

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

where $\dot{\gamma}$, η and K are the shear rate, viscosity and constant, respectively. The values of the flow behavior index, n , for 130°C, 150°C and 170°C are 0.646, 0.701 and 0.578 respectively, where all values are less than 1 at all temperatures which confirmed the pseudoplastic flow or also known as shear thinning behavior. The degree of shear sensitivity of feedstock is indicated by these values. The lower the value of n means more viscous dependence on the shear rate. Meanwhile, if the flow behavior index is greater than 1, it indicates a dilatant flow which may lead to powder-binder separation due to high shear rate.

The activation energy, E is determined based on Arrhenius equation

$$\eta = \eta_0 \exp(E/RT), \quad (2)$$

where η_0 is the viscosity at the reference temperature, R is the gas constant, and T is the temperature in Kelvin unit. Using a load of 140 N as a reference, the graph of $\ln \eta$ versus $1/T$ is plotted and shown in Figure 2.

A large value of activation energy, E means a high sensitivity of viscosity towards temperature and pressure which leads to the formation of defects during the injection molding process [11,13]. The value of E for the 72vol% powder loading is 20.91 kJ/mol, which is the lowest compared to 71vol% and 73vol% where the values are 21.02 kJ/mol and 21.07 kJ/mol, respectively. In addition, these values are considered low for PIM.

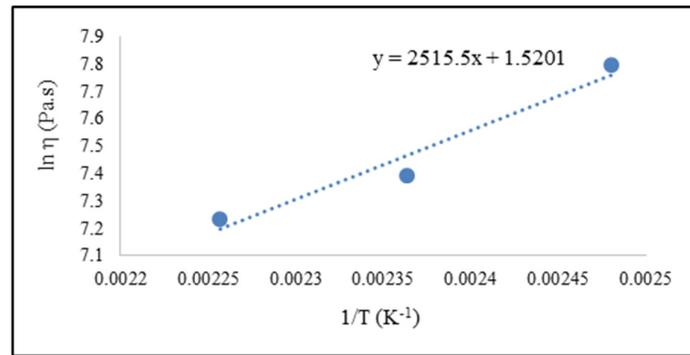


Fig. 2. Graph of $\ln \eta$ (Pa.s) versus $1/T$ (K^{-1})

3.2 Green Part

Feedstock at 72vol% powder loading is successfully injected where no defects such as flashing and short shot are observed on the surface of green part, as shown in Figure 3. The green part is successfully produced due to the sufficient values of mold temperature, melt temperature and pressure in filling the mold completely during the injection molding process [14]. It is also observed that the color of the injected parts changed to brown after the thermal debinding process is completed. It is also observed that the debound parts are still intact and will be sintered to obtain the final parts. The homogeneity of the 72vol% feedstock is proven by the SEM image as shown in Figure 4 where it is observed that the powder and binders are well mixed.

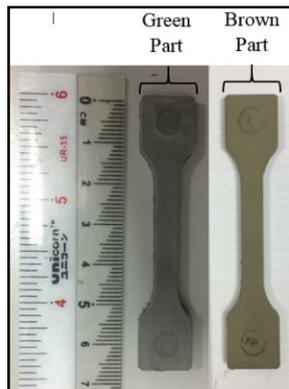


Fig. 3. Green parts and brown parts

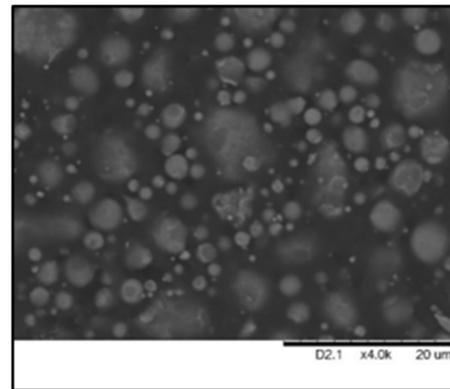


Fig. 4. Well mixed between the powder and binders for the 72vol% feedstock

Three-point bending flexural test is then conducted on the green part and the maximum strength of 5.51MPa is obtained. Such value is in the good range of green part strength for metals in PIM, which is 4-7MPa [13,15,16].

4. Conclusion

The suitability of PS and PE as binders for SS17-4PH is proven where a pseudoplastic flow type was obtained from the rheological behaviour investigation. Such type provides the suitable range of viscosity and shear rate needed in PIM. In addition, the feedstock at 72vol% powder loading gave the lowest value of activation energy, E which means such feedstock has low sensitivity towards

temperature and pressure changes. Such low sensitivity resulted to no significant defects were observed on the injected parts which also may due to the homogeneity of the feedstock. Therefore, the injectability of SS17-4PH with PS and PE binders was successfully investigated.

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