The Characterization and Rheological Investigation of Materials for Powder Injection Moulding

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ABSTRACT

Stainless steel and Zirconia have become an attractive choice of powder injection moulding (PIM) materials because of their respective excellent mechanical and thermal properties. These are often combined deliberately to produce functional graded components. The characterization and rheological investigations of the materials are thus indispensable for successful implementation of subsequent stages to the PIM process. In this investigation, the powder materials were characterized. Two feedstock with solid loadings of 68% for stainless steel (17-4PH) and 50% for 3mol% yttria stabilized zirconia (3YSZ), were prepared based on optimal loading of 3% lower than the critical value. A common binder was employed comprising of 60% palm stearin and 40% polyethylene. The rheological results for the two materials exhibited pseudo-plastic and shear thinning behaviour indicated by decrease in velocity with increasing shear rate. The results also show that a temperature of 130^{0}C is considered appropriate for injection moulding of both feedstock.

Keywords*: 2C-Powder Injection Molding, Solid Loading, Binder Characteristics, Feedstock, Rheology*

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Introduction

PIM applications will continue to expand as a result of constant development and increased demand for integration of a number of functions in many structural and functional components. The PIM process is a net shape manufacturing process developed from combination of excellent shaping capability of plastic injection moulding and flexibility of powder metallurgy [1]. The process consist of four processing steps; mixing, injection moulding, de-binding and sintering. A homogenous mixture of powder and binder termed 'feedstock' is granulated and injection moulded to produce green compact. The binder is subsequently extracted and the compact sintered to obtain the final component at near full density. In recent times, the PIM process has evolved to two component injection moulding (2C-PIM) for production of small, complex and high functional devices [2, 3]. Thus 2C-PIM involves injection of two material feedstock either simultaneously or sequentially in one moulding machine via a single processing route [4]. The integration of different functionalities in one part makes this process very attractive and economically relevant.

The powder characteristics are fundamental for successful PIM processing. It is therefore important that a precise balance is maintained between powder attributes, binder composition and ratio of powder to binder. The stainless steel (17-4PH) and zirconium (3YSZ) powder material have been selected for this study. Stainless steel (17-4PH) is widely used in PIM industries [5]. It is also used as structural materials in many applications due to excellent mechanical properties. On the other hand, 3YSZ has been extensively investigated and widely used because of its exceptional mechanical and functional properties such as high toughness, oxygen diffusivity and low thermal conductivity [6]. One of the dominant powder characteristics is the optimum solid loading which is estimated based on the critical solid loading. The critical solid loading corresponds to a composition where the particles are in point contact and the interspaces filled with binder. Moulding is usually performed at the optimum solid loading which is taken as 2-5% lower than the critical value [1].

The binder is well known to be the key component which provides formability and flow-ability required for moulding [7]. Thus, palm stearin (PS) and low density polyethylene (LDPE) binder system have been selected for this investigation. The palm stearin is known to possess good qualities of a binder. It has the ability to provide a capillary route for the removal of remaining binder during the later stages of de-binding prior to sintering [8]. The polyethylene binder system is a conventional thermoplastic binder for powder injection moulding process (PIM). The binder system constituents are generally grouped into two for injection moulding applications [9]. These consist of the low molecular weight polymer characterized by low temperature decomposition and high molecular weight polymer which possess relatively higher temperature decomposition [10]. The component PS is a low molecular weight binder and is used to wet the powder particles and provide flow-ability while LDPE has a higher molecular weight which ensures sufficient strength after injection moulding and de-binding processes. The latter is known as secondary or backbone binder while the former is the primary binder. In some cases, a binder system may contain a third component such as surfactant which improves the compatibility between powder particles and binder. However, it is known that PS plays the role of a surfactant and a lubricant [10].

The key variable describing the rheological behaviour of the PIM feedstock is viscosity. The lower the magnitude of the viscosity, the easier for the feedstock to flow and vice-versa. Thus viscosity plays a great influence on the constant flow and uniform filling of feedstock into the mould cavity. Good rheological evaluations will eliminate those conditions that cause flow instabilities during moulding, thereby preventing defects. Thus a predictable and homogenous feedstock with desirable rheological characteristic is imperative for successful implementation of the PIM process.

This research focuses on characterization and rheological investigation of selected PIM materials as a prelude to two component PIM fabrication. The characterization and investigations of rheological properties are performed on separate feedstock to establish required parameters for co-injection of the PIM materials.

Methodology

The morphology of the SS17-4PH and 3mol% YSZ powders were observed using Scanning Electron Microscope (SEM) and Field Emission Scanning Electron Microscope (FESEM) respectively and are shown in Figure 1. The SS17-4PH powder was supplied by Sandvick Technologies Ltd. and with a mean size of 22µm according to the manufacturer's specification. The 3mol% YSZ powder supplied by Nabond Technologies Co. Ltd., had a mean size of <5µm as indicated by the manufacturer. The densities of SS17-4PH and 3mol% YSZ were determined as 7.78g/cm3 and 5.96g/cm3 respectively using a Helium Pycnometer. In this investigation, the binder system used composed of 60% Palm Stearin (PS) and 40% Low Density Polyethylene (LDPE). The binder characteristics were determined using Differential Scanning Calorimeter (DSC) and Thermo-gravimeteric Analyzer (TGA). The PS was supplied by Intercontinental Fats Sdn Bhd with a density of 0.891g/cm^3. The LDPE was supplied by Titan chemicals Sdn Bhd and had density of 0.919g/cm^3. The feedstock for the two materials was prepared separately. Mixing of the feedstock was performed on a Brabender mixer at a speed of 30rpm and temperature of 140^{0}C. Homogeneity of the feedstock was obtained when the torque maintained a steady value for more than 30minutes. The dough like mixture was allowed to cool down and extracted from the mixer. The pieces of the dough were granulated in a crusher to finer sizes. A Schimadz capillary rheometer was subsequently used to evaluate the deformation behaviour of the feedstock with temperature varied from 130^0C to 150^0C.

Figure 1(a): SEM Micrograph of 17-4PH

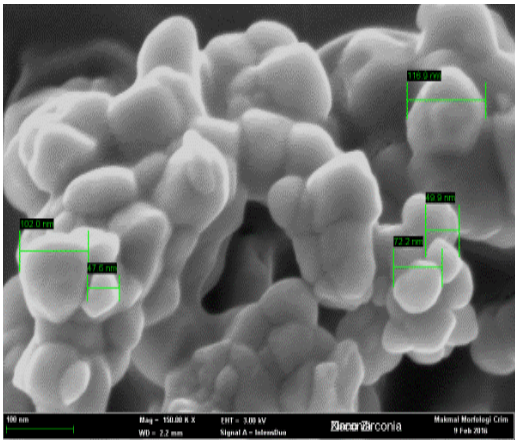

Figure 1(b): FESEM Micrograph of 3YSZ

Result and Discussion

Characteristics of the binders

From the DSC result, the melting points of the binder components are indicated by the peak temperatures corresponding to 63^0C and 117.3^0C for PS and LDPE respectively. This peak represents the temperature at which maximum heat absorption occurs. The LDPE binder shows a higher melting point and therefore mixing and moulding must be performed at a temperature above this point. On the other hand, the mould temperature must be maintained at a temperature below the lower melting point (63^0C) to avoid adherence of the moulded part to the mould cavity. Figure 2 indicates the DSC plot for LDPE and PS. Mass decomposition in relation to the temperature profiles are shown in Figure 3 for both feedstocks. The TGA provides information about the evaporative range of the binders. The data indicates that total decomposition with no residue occurs at 460^0C for PS and 500^0C for LDPE. This information is very useful for designing de-binding schedules to ensure complete binder burn out. The binder characteristics from the results are shown in Table 1.

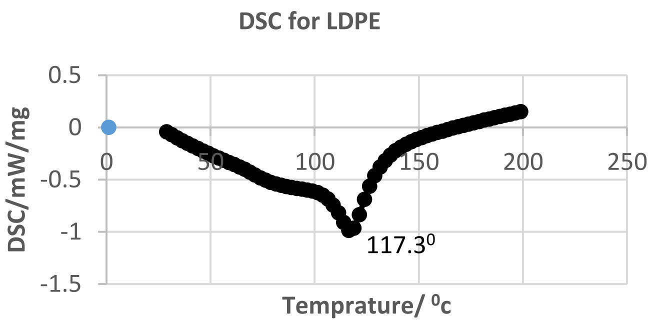

Figure 2(a): DSC plot for LDPE

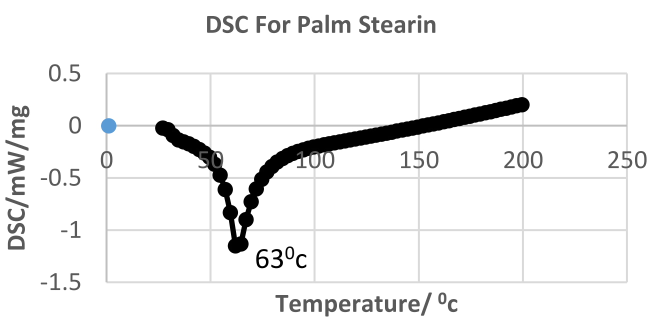

Figure 2(b): DSC plot for Palm Stearin

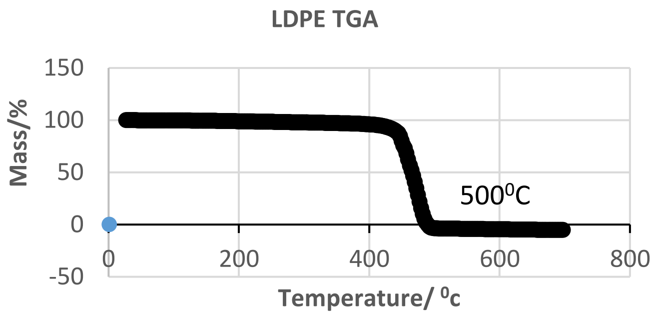

Figure 3(a): Mass decomposition versus temperature profiles for LDPE

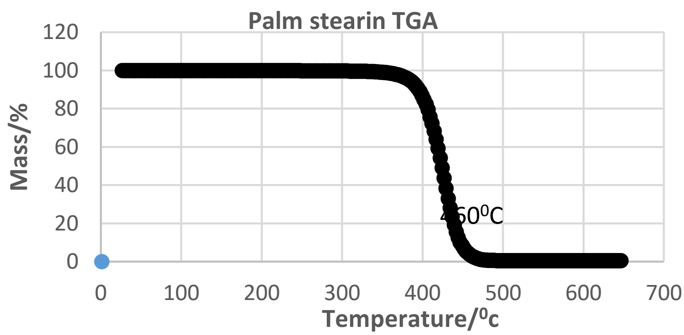

Figure 3(b): Mass decomposition versus temperature profiles for PS

Table 1: Characteristics of binders

Binder	Melting point	Degradation temperature	Density(g/cm^3)
LDPE	117.3^0C	500^0C	0.919
PS	63^0C	460^0C	0.891

Solid Loading of Powders

Solid loading have a significant influence on the rheological behaviour of the feedstock. Feedstock are usually formulated with powder slightly less than the critical value. In this investigation, a solid loading of 68% and 50% for SS17-4PH and 3YSZ powders respectively were chosen as optimal loading with each 3% less than the critical value. The powder was blended with oleic acid as a binder in a brabender mixer with a speed of 25rpm at room temperature until maximum torque is obtained. A high solid loading was preferred to minimize shrinkage during subsequent steps such as de-binding and sintering [11]. However, excessive solid loading is unacceptable because of moulding difficulties resulting from increase in viscosity. It is well known that the volume of the binder reduces as the volume of the powder is increased, thus increasing the viscosity. On the other hand, low powder loading causes large volume shrinkage especially after de-binding and sintering thus making it difficult to obtain a tight dimensional tolerance control [12] as well as powder binder separation during moulding process. Figure 4 depicts torque evolution as a function of time for determination of the critical solid loadings for SS17-4PH and 3YSZ.

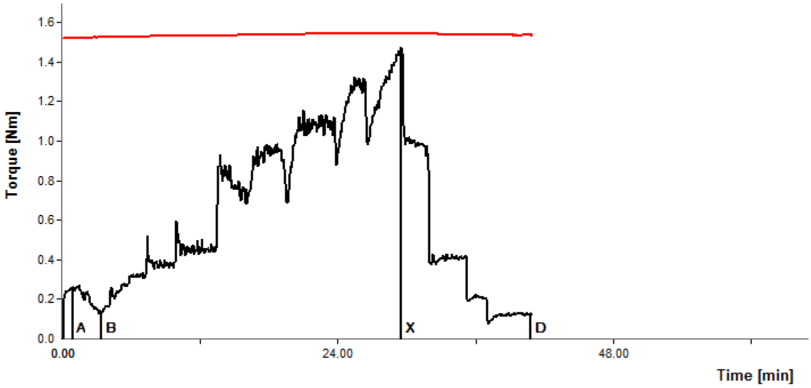

Figure 4(a): Torque evolution related to time for SS17-4PH.
Critical solid loading = 71%

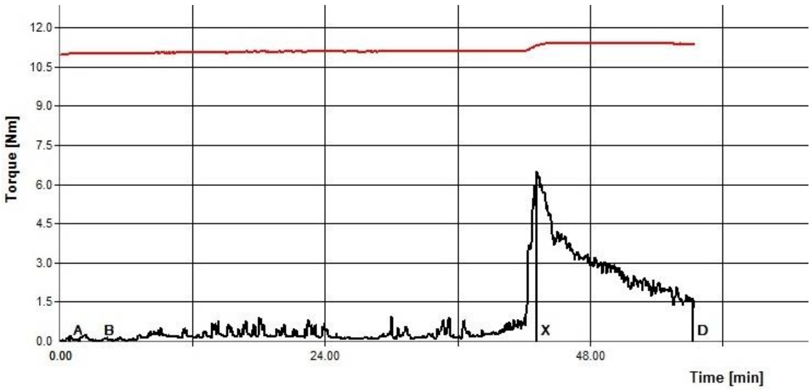

Figure 4(b) Torque evolution as a function of time for 3YSZ.
Critical loading = 53%

Feedstock formulation

In order to maintain a balanced mixture of powder and binder, the feedstock formulation was performed on weight basis applying the theoretical densities of their constituents. The weight fraction of the powders and binders were subsequently evaluated. Thus feedstock formulation is a very critical stage since defects arising from inhomogeneous feedstock such as voids, cracks or distortion cannot be reversed in subsequent processing stages. In addition to causing non-uniform shrinkage or warping in sintered parts, these defects present regions of agglomerates which tends to widen the particle distribution and decrease the viscosity [11]. During mixing of feedstock, the impact of the twin screw blades on the particles is by shearing action. The particles are de-agglomerated and re-arranged with the particles dispersed into the binder. The brabender mixer provides a good dispersion of the powder particles in the binder. The relationship between mixing torque evolution with time required to achieve homogeneity of the feedstock are shown below in Figure 5 for both feedstock.

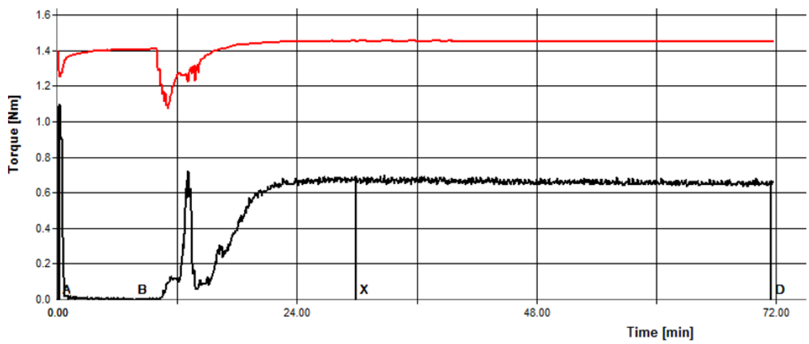

Figure 5(a): Mixing torque evolution as a function of time for SSI7-4PH

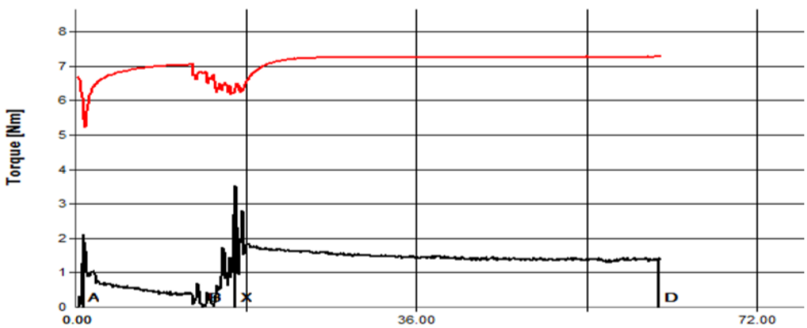

Figure 5(b): Mixing torque evolution versus time for 3YSZ

Rheological properties

The rheological properties of the feedstock are evaluated based on the temperature dependent viscosity profiles versus shear rates. Appropriate range of shear rates are usually between 10^2 - $10^5 s^{-1}$ while maximum viscosity is 10^3 Pa.s. Rheological results for the two feed stocks indicate pseudo-plastic behaviour (shear thinning, n<1), where viscosity decreases with increase in shear rate (see Figure 6). The reason may be due to particle alignment or binder molecule orientation and streamlining with flow [1, 13]. The flow behaviour index (n) indicates the sensitivity of viscosity to shear. Feedstock with low value of (n) exhibit minimum changes of viscosity by variation of shear rate during injection moulding [14]. However, extremely low value of n results in viscosity drop at high shear rate regions and is related to moulding defects such as jetting and powder-binder separation. Thus a temperature of 130^0C is selected for injection moulding of the feedstock (see Table 2). The activation energy (E) indicates the sensitivity of the viscosity to temperature. Thus a high value of E means that any small temperature fluctuations results in sudden viscosity change and vice versa.

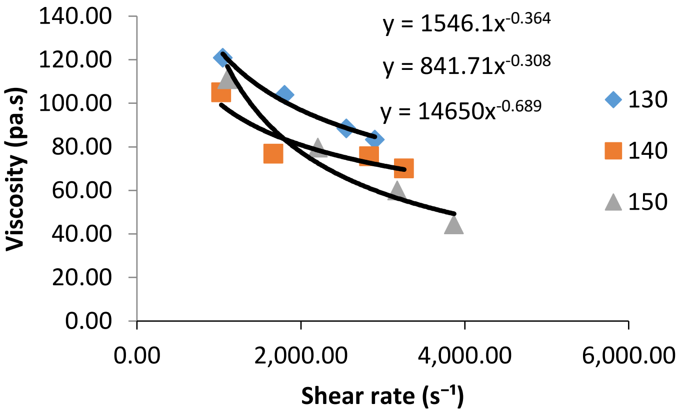

Figure 6(a): Rheological behaviour of SS17-4PH

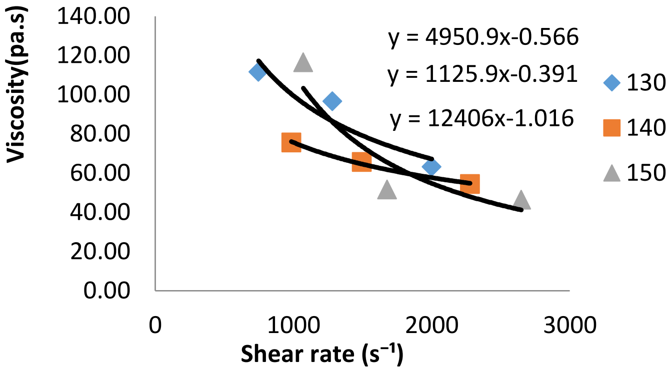

Figure 6(b): Rheological behaviour of 3YSZ

Table 2: Rhological data

Material	Power loading	Temp. (ºc)	Flow Index (n)	Material	Powder Loading	Temp. (ºc)	Flow Index (n)
SS17-4PH (E=3.7)	68%	130 140 150	0.64 0.70 0.32	3YSZ (E=154)	50%	130 140 150	0.44 0.61 -0.01

Conclusion

The characterization and rheological investigations of the PIM materials were successful. The result demonstrated that the powder loadings of 68% and 50% for SS17-4PH and 3YSZ respectively exhibited considerable rheological behaviour desirable for injection moulding for the range of temperatures. It further shows that a temperature of 130^0C is suitable for moulding. From the foregoing, it is important to note that improper characterization and formulation of feedstock inevitably lead to irreversible defects in the final component. A well formulated feedstock will have good rheological properties required for moulding including desired mechanical properties after de-binding and sintering.

Acknowledgement

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