Solvent Debinding Variables on Leaching Fat, Oil and Grease (FOG) Derivatives of Green Part Stainless Steel SS316L Metal Injection Moulding

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Abstract— Sewage fat or Fat Oil Grease (FOG) derivatives for binder’s component in the stainless steel SS316L feedstock being injected and undergo several test debinding variables. The influences of temperature, time and solvents type has been tested with binder formulation of 60:40 between Polypropylene (PP) and FOG derivatives besides the 60% powder loading of stainless steel powder. Experimental results of the quickest and higher percentage of FOG derivatives leach out from green part is being analysed. The green part will undergo two different solvent debinding (Heptane and Hexane) with three different solvent temperature. Debinding times was set for 10 hours and every 1, 3, 6 and 10 hours the green part will undergo weight measurement for monitoring the weight loss percentage of green part. It seems that both solvent indicates good diffusion and dilution for extracting the FOG derivatives out from the green part in producing brown part where no sign of part crack or swelling during and after solvent debinding. Energy Dispersive Spectrometer (EDS) for element detection and Scanning Electron Microscope (SEM) analysis also being perform which indicates the good pores build up. Results found that the best solvent for removing FOG derivatives is hexane with 60oC being the choice of solvent temperature.

Index Terms— Solvent debinding; Metal injection moulding; FOG derivatives; and binder ratio.

I. INTRODUCTION

Metal injection moulding (MIM) is a process with an advantage of producing complex, intricate and near-net-shape parts in highly automated with high volume production in a few shot as compare to other fabrication process[1]–[3]. After being injected, the parts will undergo debinding process and finally sintering process. 316L stainless steel is one of the most widely used materials for MIM research and industrial applications. Since 316L stainless steel is a highly alloyed material, the mechanical and corrosion properties are good and recognised as material for implants in medicals arena.

The viscosity along with shear rate plays an important property to flow behaviour during mould cavities filling stage. Understanding the rheological behaviour and influences of the parameters of injection mould process is necessary since it will contribute to the strength and density of the green part[4], [5]. Proper injection parameters need to be determined since it will influence the outcomes of the green part due to the feedstock will experience high shear during this states. Too high in injection pressure could cause powder binder separation and segregation which leads to inhomogeneities during particles packing[6]. Improper injection parameters also could cause only plastic will flow inside the mould cavity and tend to leave the powder inside the barrel.

After proper injection process parameters being selected, the samples will undergo solvent debinding where here the secondary binder will be removed through the method of diffusion, dissolution, dilution and leach out from the sample. The removal of organic binder is a procedure that is known to be critical in obtaining a green compact with desired microstructure[7]. Debinding is a key problem with PIM
because the time for binder removal depends on the section thickness[8]. Debinding rate were influenced by shape factor defined as ratio of volume to surface area of the sample[9]. Solvent debinding usually applied as two step debinding procedure where the extraction or leaching of the low molecular weight constituents binder will creates an open pores for degradation of the insoluble binder components to diffuse to the surface easily. The integrity of the green part must remain tight without experience any crack or swell during thermal debinding which results from internal stress built up from the inside of the parts. With high porosity created from the solvent debinding, will results in thermal debinding could be done in much shorter time and the combination of both could generate time saving of debinding process[10]. Consequently, various manufactures have set upper limits on section thickness ranging from 10 to 50 mm[8]. Thus, the goal in debinding is to remove the binder in the shortest time with the least impact on the compact [10].

Here, solvent debinding was done in leaching FOG from green part. FOG was used as secondary binder since it will promote sustainability in manufacturing area as reported in Brandtland report[11]. With increasing of human population and changing of a lifestyle towards diet, peoples tends to create more FOG and this could be the opportunity for recycling the FOG into useful materials besides making the environments better.

II. EXPERIMENTAL RESULTS

A. Materials

Polypropylene (PP) and FOG derivatives of 60:40 percentage weight ratio was used. Water atomized stainless steel 316L powder having irregular in shape with mean size d50 6 µm and tap density of 8.0471 g/cm³ supply by Atomix Epson Japan were used. Table 1 and Fig. 1 shows the properties of the binder constituent and particles size distribution of the water atomised SS316L.

Table 1: Weight percentage and thermal properties of binder constituents with the same 60% powder loading

<table>
<thead>
<tr>
<th>Binder</th>
<th>wt%</th>
<th>Melting temperature (°C)</th>
<th>Degradation Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polypropylene, PP (%)</td>
<td>60</td>
<td>165</td>
<td>358</td>
</tr>
<tr>
<td>FOG derivatives (%)</td>
<td>40</td>
<td>50</td>
<td>274</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Injection Temp, A (°C)</th>
<th>Injection Press, B (MPa)</th>
<th>Mold Temp, C (°C)</th>
<th>Cool Time, D (s)</th>
<th>Injection Speed, E (cm/s)</th>
<th>Injection Time, F (s)</th>
<th>Packing Time, G (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>190</td>
<td>80.5</td>
<td>60</td>
<td>10</td>
<td>10</td>
<td>4</td>
<td>3</td>
</tr>
</tbody>
</table>

Table 2: Parameters setting for injection moulding machine

Degradation temperature of both binder constituents was found to be 358.8°C and the lowest is 274.1°C[12]. Degradation temperature is needed to be known here since mixing temperature should not exceed the lowest degradation temperature which results in losing the capability of one of the functions of binder components.

B. Injection Moulding

Water atomised stainless steel feedstock was injected using Nissei NP7-1F with parameters setting shown in Table 2 after being optimised using Taguchi method. Mixing of the feedstock was done for 1 hour before being crush and used for injected into a sample[12]. Figure 2 shows the green part of the sample after being injected.

C. Solvent debinding process

Solvent leaching operations were carried out by using different organic solvents (n-hexanes, n-heptanes) at different ranging temperature from 40°C to 60°C (Fig 3). Another fixed variable implemented in this experiments is solvent feed ratio where
here 14:1 solvent feed ratio being chosen since higher than 7:1 solvent feed ratios will not give any significant effect to debinding process[8]. Time of leaching process will be done up to 10 hrs. Experiments for all samples and temperatures and time were run in duplicate.

Diffusion coefficient against time (Fig.4) being plotted base on Eqn. 1.

\[
\frac{C_a - C_0}{C_l - C_0} = \frac{8}{\pi} \frac{\pi}{h^2} \exp(-\frac{\pi^2 D t}{h^2})
\]

(1)

Where \(C_a\) is the average concentration of binder remaining on compressed part, \(C_l\) is the initial concentration of binder, \(C_0\) is the debinding condition (zero), \(t\) is the debinding time, \(D\) is the debinding coefficient and \(h\) is the thickness of the compressed part (Nanjo et al., 1993 cited in [13]) which was taken from Fick’s 2nd Law.

![Fig. 3: (a) the samples were placed inside the beaker using wire stand (b) Samples kept submerge into solvent (c) Samples being placed inside the oven at setting temperature.](image)

The debinding or weight loss percentage were calculated according to the following equation;

\[
\% \ W_{\text{loss}} = \frac{(W_{\text{initial}} - W_{\text{after}})}{W_{\text{initial}}} \times 100
\]

(2)

Where \(W_{\text{initial}}\) is the weight of compressed bodies or green compact and \(W_{\text{after}}\) is the weight of green compact after solvent debinding.

![Fig. 4. Diffusion coefficient of the hexane and heptane solvent at 60 °C toward the green compact](image)

**III. RESULTS AND DISCUSSIONS**

**A. Leaching Temperature**

![Fig. 5. Shows the debinding curve of weight loss with different temperature with respect to time for solvent (a) n-Hexanes (b) n-Heptanes](image)

The solvent extraction debinding process is considered the most important step among many steps in MIM. Dilution and diffusion of suitable solvent in binders is frequently used as a means of accelerating the removal of the constituents of the binder system while leaving the backbone binder to support the structure. This backbone binder is removed later by a thermal treatment which, because of the porosity network created by the solvent extraction step, can performed rapidly [8].

Solvent temperature is one of the important variables in leaching processes. Effect of temperature on the leaching processes can be seen in Fig.5 for both hexanes and heptanes solvent with solvent to feed ratio of 14:1 with duration time of
10 hours. As the temperature increase the percentage weight loss of binder constituents also increase for both solvents. This attribute that the 60°C solvent temperature is the fastest rate of leaching out the FOG as compare to others temperature. Although the highest temperature will accelerate the extraction of the binder constituents, defects such as surface cracks, bloating, swell, blistering can be occurred due to high dilution and diffusion of the solvent which results in quick expansion of the binder which results in high internal stresses[8]. From the results no defects were found on the samples although the temperature were set at 60°C (Fig. 2). Thus the 60°C solvent temperature were chosen as a suitable leaching temperature for extracting FOG of the binder system.

It can be noticed that in Fig. 4 that hexane solvent gives higher diffusion rate as compared to heptane. It is noticeable that amount of binder extraction increase with lower carbon number of the solvent[8]. Lower carbon numbers means lower viscosity of the solvents and easy penetration and diffusion of the FOG from the green compact[8]. The highest diffusion rate at 60°C temperature after 1 hour is hexane. The diffusion rate is decreasing with respect to time due to less concentration of FOG remaining after several hours which results in decreasing of diffusion coefficient[8].

B. SEM/EDS and DSC Analysis

Results from Scanning Electron Microsturcture (SEM)/Energy Dispersive Spectroscopy(EDS) analysis shown in Fig. 6 to confirm that as the time of debinding were increases the pores and particles shape become clear with sign of PP polymer attach to the particles due to most of FOG being extracted from green compact.

Moreover it can be notice from Differential Scanning Calorimeter (DSC) analysis by comparing the green compact (Fig. 7(a)) and brown part after 10 hrs solvent debinding (Fig. 7(b)), the FOG derivatives melting peak disappear which indicates complete removal of FOG during the process.
C. FTIR analysis

FT-IR stands for Fourier Transform InfraRed, the preferred method of infrared spectroscopy. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. Fig 8 shows the analysis of the green part as compare to PP copolymer and also FOG derivatives[14], [15]. As can be seen that FOG derivatives has several characteristic between regions of 1500cm\(^{-1}\) to 3000cm\(^{-1}\). In Fig 8(a), the peak value of the 2917cm\(^{-1}\), 2849cm\(^{-1}\) and 1706cm\(^{-1}\) seem to match with the peaks obtained from the green compact which indicates the existence of FOG inside the green compact. Fig. 8(b) represents the PP polymer inside the green compact. The characteristic of PP are between the regions of 2000cm\(^{-1}\) and 3300cm\(^{-1}\). The presence of PP can be match with the peak of 2170, 3257and 3250 with the peak obtained from the green compact. This indicates that the FOG and PP are well intact with the stainless steel powder. Results from this analysis can be used to represent binder composition remaining inside the polymer but heptane’s indicates some portion of FOG as can be seen in wavelength of 2849cm\(^{-1}\) which results in much easier of dissolution of FOG.

IV. CONCLUSIONS

Solvent debinding of green part using both hexanes and heptane solution were successfully removed FOG derivatives. Results from the EDS analysis shows significant decrement in carbon element. FTIR results also indicates hexanes solution become the better choice of solvent for extracting the FOG beside the analysis of debinding rate and weight loss performance analysis with both solution. This is due to the low viscosity of the hexanes as compare to heptane which results in easy penetration of hexanes solution inside the green compact.

REFERENCES


