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Particle Size and Injection Temperature Effect on the Injection Molding of SS316L Powder

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ABSTRACT

Particle size and injection temperature influence on the injection of SS316L powder has been investigated. The feedstocks used for the investigation being 64 vol. % and 65 vol. %. Results found that the success of the molding process depends on the feedstock rheological properties. Fine powder particles at 64 vol. % demonstrated higher temperature sensitivity than the coarse powder feedstock. However, the coarse powder feedstock shows its sensitivity to the injection pressure. The investigation found that the injection temperature has its influence to the final quality of the compact. Injection temperature of 140°C was found to be the optimum. However, the investigation does not found any significant on the injection temperature to the debinding rate.

Keywords: *Metal injection molding, particle size effect, as-molded, water leaching.*

Introduction

Metal injection molding (MIM) has emerged as a viable method of producing complex shaped parts at a competitive cost [1]. The MIM process uses a combination of powder metallurgy and injection molding technologies to produce net-shape parts and is comprised of five main sub processes: raw materials selection (powder/binder), feedstock preparation, injection molding, debinding, and sintering.

One of the advantages of powder injection molding is its ability to produce parts with complex geometry without machining. However, to stay within the ever-tighter tolerances demanded by component manufacturers' customers, MIM parts have to be produced with a high degree of dimensional control in order to minimize the dimensional variability of critical dimensions [2, 3].

Zauner et al. [4] investigates the effects of powder type and powder size on dimensional variability. Powder characteristics play an important role in the MIM process thus Zauner et al. [4] conducted a study to show the effect of powder size, powder shape, powder size distribution, and surface area focusing on dimensional variation and its dependence on powder type, particle shape, and particle size. Arakida and Miura [5] studied fine (20 μm), coarse (150 μm) gas- and water-atomized powders, and their effect on packing density and fluidity. Their work showed that fine gas-atomized powder exhibits higher density, which in turn produces improved mechanical properties. Dihora et al. [6] found that the instability index for feedstocks increases with particle size. Almost all of the studies that dealt with powder characteristics focused on rheometry rather than dimensional variation.

Yimin et al. [7] have investigated effect of powder loading (60, 64, 68 and 72 vol. %) on MIM stainless steel. The investigation proved that 68 vol. % powders loading was the optimum for injection molded gas atomized spherical 17-4 PH stainless steel powder and the binder of 65% PW + 30% EVA + 5% SA. The 68 vol. % powder loading can be injection molded with a comparatively low viscosity on a relatively wide temperature range, and it is best to get quick powder re-packing and binder molecule orientation during injection molding. From the standpoint of compact shape retention and dimension tolerance control, the optimal powder loading of 68 vol. % was also the best. Furthermore, the compact of 68 vol. % powder loading is easy to get sinter densification and has superior mechanical properties and microstructures.

While, in another work, Hwang et al. [8] investigated the debinding rate of solvent debinding for compacts prepared with different particle sizes. The investigation concluded that the debinding rate is determined by the cross-section thickness, particle size does not affect the torturosity for spherical powders, and thus not the debinding rate. Further, on the investigation to higher powder loading compacts, the debinding rate was slightly slower. With a higher powder loading, the debinding rate decreases because the total porosity and flux area for the soluble binder component to diffuse through decreases. This paper aims to present authors' work on the investigation of the temperature influence on the injection of 316 L coarse and fine powders feedstock.

Experimental Procedures

Materials

The metal powder used in this study is the ANVAL 316L stainless steel gas atomized powder with the pynometer density of 7.93 g/cm^3. A binder system based on polyethylene glycol (PEG) was prepared. The minor component is polymethyl methacrylate (PMMA) and stearic acid (SA) was added as the surface-active agent. The binder composition is 73% PEG + 25% PMMA + 2% SA based on the weight fraction and the powder loadings are 64 and 65 vol. %.

The distribution of particle size as shown in Table 1 was measured using Mastersizer, Malvern Instrument. This method was used to measure the size percentage of the powder.

Table 1: Particle Size Distribution

	D_{10}	D_{50}	D_{90}	S_w
Coarse	9.563	19.606	40.058	4.159
Fine	5.780	11.225	19.840	4.873

Experiment Procedure

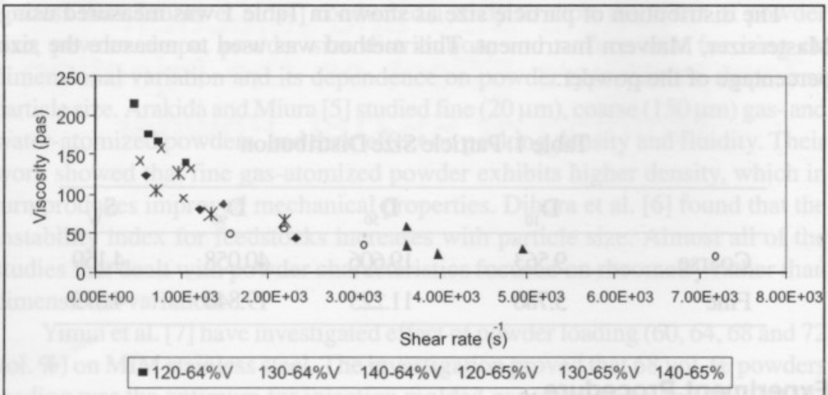
Prior investigation, stainless steel powder was mixed with binders in the sigma blade mixer for 95 minutes at 70 ºC. After mixing, the paste was removed from the mixer and will be fed into the strong crusher for granulation.

The rheological characteristic of the feedstocks were investigated using Shimadzu 500-D capillary rheometer and the MIMA tensile specimen was injection molded with the Battenfeld BA 250 CDC injection-molding machine. In order to evaluate the temperature influence, injection pressure was remains at 350 bars while the injection temperature was varied from 120, 130,140 and 150ºC.

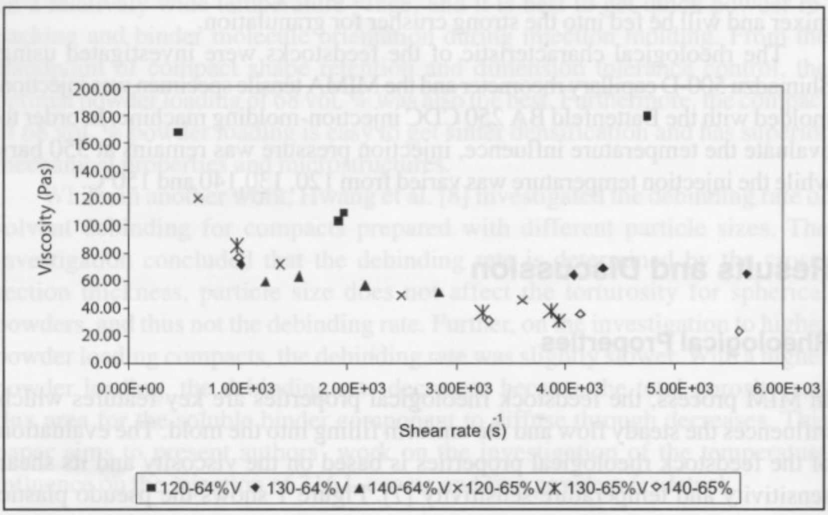
Results and Discussion

Rheological Properties

In MIM process, the feedstock rheological properties are key features which influences the steady flow and the uniform filling into the mold. The evaluation of the feedstock rheological properties is based on the viscosity and its shear sensitivity and temperature sensitivity [7]. Figure 1 shows the pseudo plastic behavior of both feedstocks at injection temperature of 120, 130 and 140ºC. Generally, the viscosity was decreasing when shear rate was increased.



(a) fine powder



(b) coarse powder

Figure 1: Feedstock Pseudo Plastic Behavior

Figure 1 (a) shows the fine powder feedstock was more viscous than that shown in Figure 1 (b). This is due the fine powder contains smaller interstitial spaces than the coarse powder thus it increase its interparticle friction. Beside that, fine powder has larger particle surface contact area between powder particles [1].

An MIM feedstock is generally considered pseudo plastic fluid [9]. For pseudo plastic fluid, there is

$$\tau = k\left(\dot{\gamma}\right)^{n} \quad (1)$$

where t is the shear stress, γ the shear stress, k the constant and n is the flow behavior index (<1). The value of n indicates the degree of shear sensitivity. The lower the value of n, the more quickly the viscosity of feedstock changes with shear rate. Injection molding of MIM feedstock is conducted under pressure and temperature. It is desirable that the viscosity of the feedstock should decrease quickly with increasing shear rate during molding. This high shear rate sensitivity is especially important in producing complex and delicate parts.

Table 2: Activation Energy and Flow Behavior Index

Abbreviation	E (kJ/mol)	Temp (ºC)	n
16_64 (fine)	79.54	120	0.49
		130	0.23
		140	-0.22
16_65 (fine)	33.67	120	0.67
		130	0.49
		140	0.36
31_64 (coarse)	49.33	120	0.98
		130	0.95
		140	0.81
31_65 (coarse)	9.8	120	0.43
		130	0.33
		140	0.35

Moreover, the feedstock viscosity depends exponentially on absolute temperature T as follows [9]:

$$\eta = \eta_o \exp\left(\frac{E}{RT}\right) \quad (2)$$

where η_o is the reference viscosity, E is the flow activation energy, R the gas constant and T is the absolute temperature. The value of E expresses the effect of temperature on the viscosity of the feedstock. If the value of E is low, the viscosity is not so sensitive to temperature variation. Therefore, any small fluctuation of temperature during molding will not result in sudden viscosity change. A sudden viscosity change could cause under stress concentrations in molded parts, resulting in cracking and distortion [9, 10, 11].

Table 2 shows the activation energy at shear rate 1000 s^{-1} and flow behavior index of the feedstock. Fine powder feedstock exhibits higher sensitivity than the coarse feedstock. This can be seen that the fine feedstock has high activation energy than the coarse feedstock. However, Table 2 shows that the sensitivity was reducing when the powder loading is increases.

Further, the flow behavior indexes (< 1) demonstrate a shear thinning of the feedstock when applied to shear stress. The flow behavior indexes of each feedstock are reducing when the injection temperature arises. Lower the flow behavior index, higher the sensitivity. As 16_64 has negative flow behavior index at 140°C, then it shows that this feedstock is very much sensitive to the change of temperature and shear rate during injection. The melt will possibly to freeze inside the mold cavity when drastic temperature change occurs in the mold cavity.

Therefore, any small fluctuation of temperature during molding results in a sudden viscosity change. This could cause defects in the molded parts, such as cracking and distortion due to the undue stress concentration appeared [10]. In addition, feedstock with high sensitivity to temperature is also sensitive to pressure [1].

As Molded

After the green compact has been ejected from the mold cavity, then the green strength of each compact produced were tested with the three-point bend test using INSTRON 5567 according to the MPIF Standard 15. The density was measured with Archimedes water immersion method according to the MPIF Standard 42. The result is as shown in Figure 2.

Figure 2 shows that in overall the as-molded strength were increasing when the injection temperature was increased. The plot shown that 16_65 was stronger than 16_64 at 120 and 130°C but was vice versa at 140°C and 150°C. At most of injection temperatures, 31_65 was the strongest except at 120°C. This is because at high powder loading feedstock has ability to increase the as-molded strength because powder particles interlocked each other's [1].

Figure 3 shows the as-molded density at various injection temperatures. 16_65 has higher as-molded density than the others do at all injection temperatures followed by 31_65. In general, the result shows that the fine powder feedstock produces higher green compact density than the coarse powder as the fine powder has small interstitial spaces [1]. The result shows that 16_64, 16_65 and 31_65 were best injection molded at 140°C. This is due to the density shown in Figure 3 were at peak when it was molded at 140°C. However, the peak for 31_64 was at 150°C.

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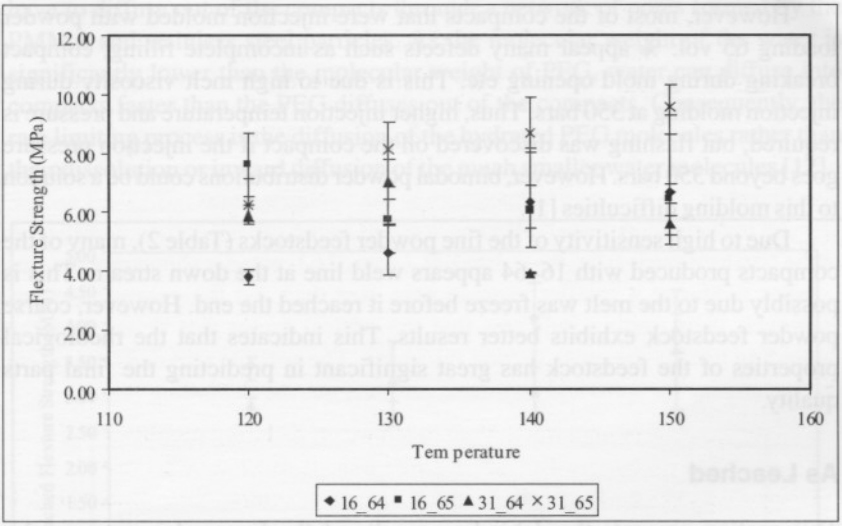


Figure 2: As-molded Strength

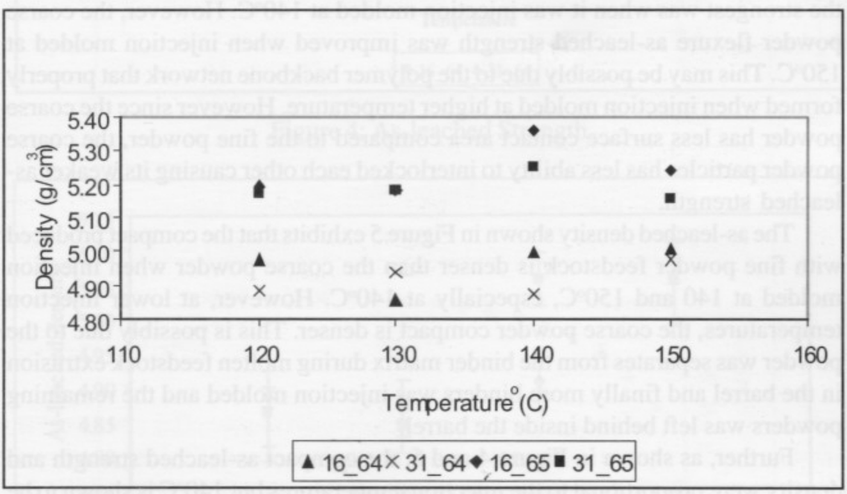


Figure 3: As-molded Density

However, most of the compacts that were injection molded with powder loading 65 vol. % appear many defects such as incomplete filling, compact breaking during mold opening etc. This is due to high melt viscosity during injection molding at 350 bars. Thus, higher injection temperature and pressure is required, but flashing was discovered on the compact if the injection pressure goes beyond 350 bars. However, bimodal powder distributions could be a solution to this molding difficulties [1].

Due to high sensitivity of the fine powder feedstocks (Table 2), many of the compacts produced with 16_64 appears weld line at the down stream. This is possibly due to the melt was freeze before it reached the end. However, coarse powder feedstock exhibits better results. This indicates that the rheological properties of the feedstock has great significant in predicting the final parts quality.

As Leached

This section presents the debinding results of the fine and coarse powder feedstocks at powder loading 64 vol. %. Three-point bend test result shown in Figure 4 is the as-leached strength of the compact. Result shows that the fine powder gives higher as-leached strength compared to the coarse powder and, the strongest was when it was injection molded at 140°C. However, the coarse powder flexure as-leached strength was improved when injection molded at 150°C. This may be possibly due to the polymer backbone network that properly formed when injection molded at higher temperature. However since the coarse powder has less surface contact area compared to the fine powder, the coarse powder particles has less ability to interlocked each other causing its weaker as-leached strength.

The as-leached density shown in Figure 5 exhibits that the compact produced with fine powder feedstock is denser than the coarse powder when injection molded at 140 and 150°C, especially at 140°C. However, at lower injection temperatures, the coarse powder compact is denser. This is possibly due to the powder was separates from the binder matrix during molten feedstock extrusion in the barrel and finally more binders was injection molded and the remaining powders was left behind inside the barrel.

Further, as shown in Figure 4 and 5, the compact as-leached strength and density were proportional to the injection temperatures but 140°C is shown to be the best injection temperature because it enables to produce stronger and denser as-leached parts. This has its significant because high density compact will minimize sintered part shrinkage.

The relation between PEG loss and immersion time in water is shown in Figure 6. During water debinding, water diffuses into the binder to react with and dissolve the PEG. PEG molecules become hydrated before they separate from the unhydrated mass to dissolve into water. The hydrated molecules of PEG then have to diffuse out of the compacts through a network of pores formed by the PMMA and stainless steel particles. As the molecular weight of the water is significantly lower than the molecular weight of PEG, water can diffuse into compacts faster than the PEG diffuses out of the compacts. Consequently, the rate limiting process is the diffusion of the hydrated PEG molecules rather than their dissolution or inward diffusion of the much smaller water molecules [12].

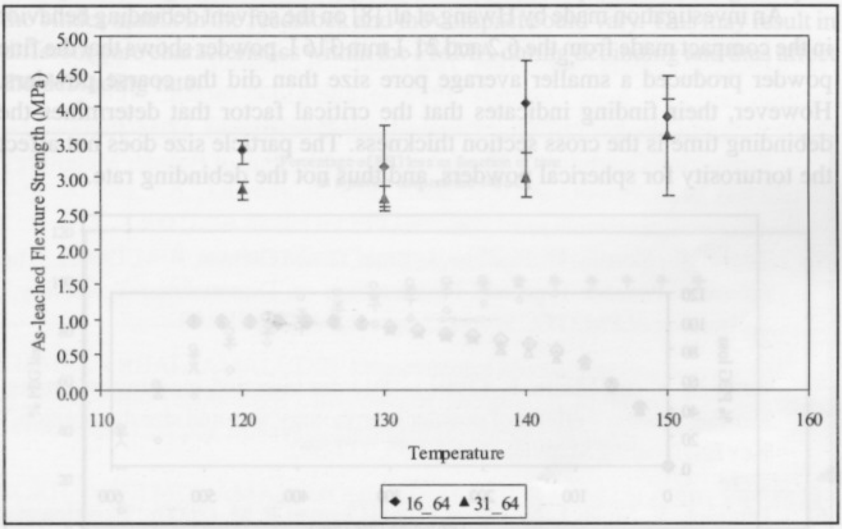


Figure 4: As-leached Strength

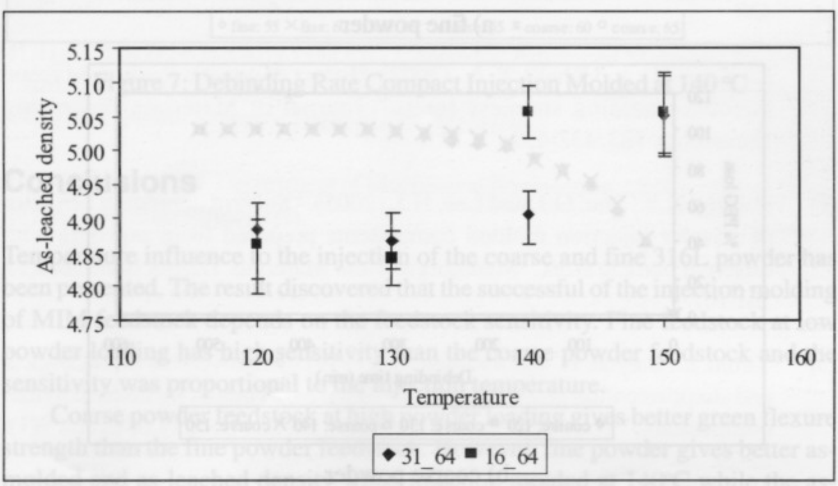
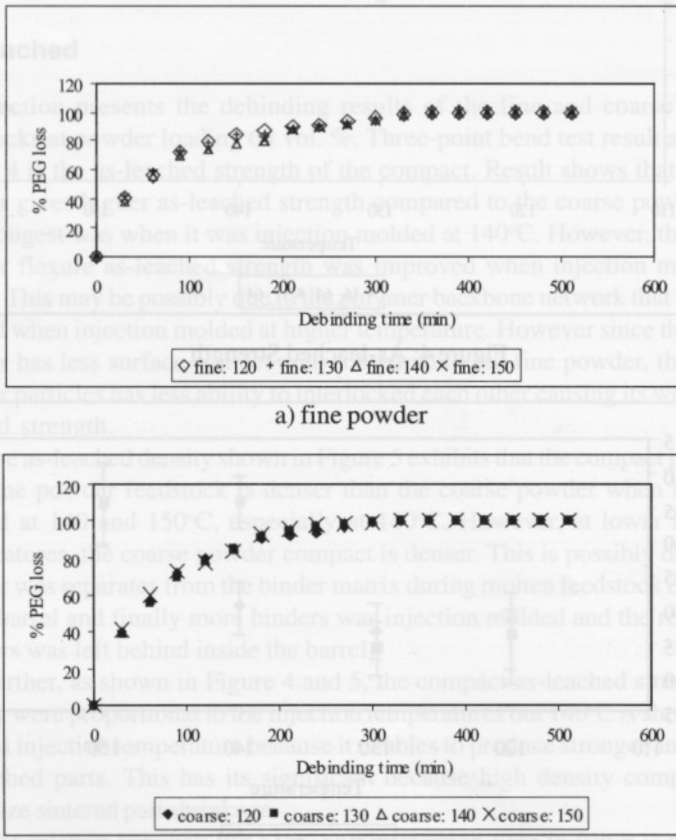


Figure 5: As-leached Density

Since the diffusion distance for the water and PEG is short in the initial stage, the debinding rate is quite fast. As debinding proceeds, however, the pore channels extend to the inner region of the compact, and the longer diffusion length slows down the debinding rate [13].

The compacts that were injection molded at different temperatures were water bath at 65°C. Results shown in Figure 6 (a) and 6 (b), does not indicate any significant of the injection temperature to the debinding rate.

An investigation made by Hwang et al. [8] on the solvent debinding behavior in the compact made from the 6.2 and 21.1 mm 316 L powder shows that the fine powder produced a smaller average pore size than did the coarse powders. However, their finding indicates that the critical factor that determines the debinding time is the cross section thickness. The particle size does not affect the torturosity for spherical powders, and thus not the debinding rate.



a) fine powder

b) coarse powder

Figure 6: Debinding Rate at 65 °C with Different Injection Temperatures (120, 130, 140, 150 °C)

However, as shown in Figure 7 the coarse powder feedstock debound faster when water bath at 65 and 60°C. The same phenomena was noticed by Hwang et al. [8], but in his investigation, smaller particles debound faster. However, in different investigation presented in the same literature, indicates, though less pronounced, an opposite trend. It is possible that the differences of debinding rates could be caused by the interaction between the powder and the binder during kneading. With different particle sizes and shapes, the homogeneity of the binder matrix in the feedstock and the compact could vary. This may result in different pore characteristics within the PMMA during debinding and thus affect the debinding rate.

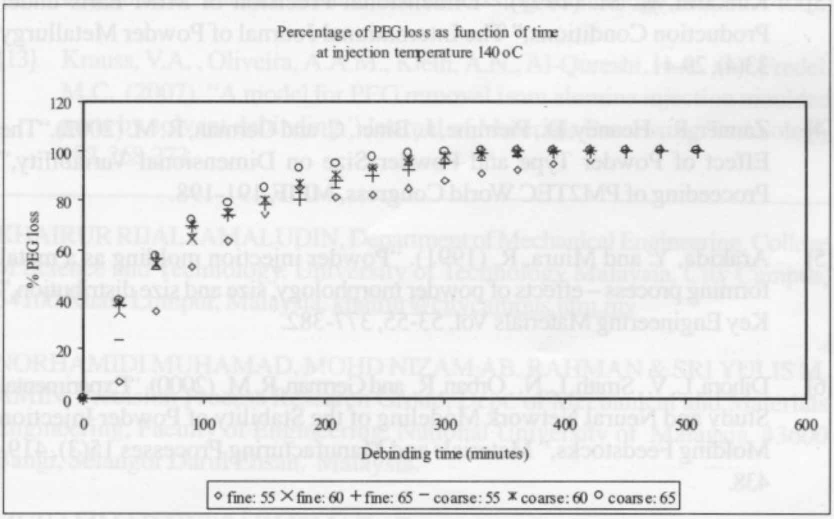


Figure 7: Debinding Rate Compact Injection Molded at 140 °C

Conclusions

Temperature influence to the injection of the coarse and fine 316L powder has been presented. The result discovered that the successful of the injection molding of MIM feedstock depends on the feedstock sensitivity. Fine feedstock at low powder loading has high sensitivity than the coarse powder feedstock and the sensitivity was proportional to the injection temperature.

Coarse powder feedstock at high powder loading gives better green flexure strength than the fine powder feedstock. However, fine powder gives better as-molded and as-leached density when injection molded at 140°C while the as-leached flexure strength was also high. Finally, the investigation found that the injection temperature does not have any influence on the water-debinding rate.

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