Powder Injection Molding an Alternative Method in the Manufacturing of Parts for Vehicles

Injekcijsko brizganje prahu, alternativna metoda za proizvodnjo delov za vozila

Joamin Gonzalez-Gutierrez [1], Igor Emri [1,2] *

[1] Faculty of Mechanical Engineering, University of Ljubljana, Ljubljana, Slovenia
[2] Institute for Sustainable Innovative Technologies – iSIT, Ljubljana, Slovenia

E-Mails: joamin.gonzalez@fs.uni-lj.si, igor.emri@fs.uni-lj.si

* Author for correspondence

Abstract: Powder injection molding (PIM) is one of the most versatile methods for manufacturing small complex shaped components from metal, ceramic powders for the use in many applications. Interesting applications of PIM include the automotive and aerospace industries. PIM can be used to combine multiple parts into a single part with complex geometry, and parts are lighter since porosity remains in the final part while the fatigue resistance increases. Therefore, it can be said that PIM can help reduce the weight of moving vehicles, which is a major concern from the environmental and economic point of view. One of the main limitations of current PIM technology is the long debinding time, which is one of the steps within the process. This has been partially solved by introducing binder systems that undergo catalytic sublimation. However, current catalytic binders have the main problem of high viscosity. In this study, two ways to decrease the viscosity of PIM feedstock materials with polyoxymethylene were investigated. The first way was to reduce the average molecular weight of the binder and the second one to select a polydisperse particle size distribution with high maximum packing fraction.

Key words: powder injection molding, viscosity, feedstock, packing fraction.

Povzetek: Injekcijsko brizganje prahu (PIM) je eden izmed najbolj vsestranskih načinov za proizvodno majhnih, kompleksnih delov iz kovinskega ali keramičnega prahu. Ena izmed zanimivih aplikacij PIM-a je vesoljska industrija, saj se PIM lahko uporablja za združevanje več delov v skupen del s kompleksno geometrijo. Z uporabo PIM tehnologije lahko torej dosežemo manjšo poroznost in povišano odpornost na utrujanje končnih izdelkov. Iz tega stališča je mogoče reči, da se lahko PIM pomaga zmanjšati težo vesoljska vozila, kar predstavlja velik problem z okoljskega in ekonomskega vidika v tej industriji. Eden izmed glavnih omejitev sedanje PIM tehnologije je čas potreben za odstranjevanja veziva, ki je eden izmed korakov v procesu. To je delno rešeno z uporabo veziva iz polioksimetilena, ki katalitično sublimira. Slaba stran uvedbe tega veziva pa je povišana viskoznost. V sklopu tega prispevka raziskujemo dva načina za zmanjšanje viskoznosti PIM surovine na osnovi POM-a. Prvi način je zmanjšati povprečno molekulsko maso veziva in drugi, da izberemo polidisperzno porazdelitev velikosti delcev z visokim deležem gostote pakiranja.

Ključne besede: injekcijsko brizganje prahu, viskoznost, surovina, gostota pakiranja.

1. Introduction

Powder injection molding (PIM) is a technology for manufacturing complex, precision, netshape components from either metal or ceramic powder. The potential of PIM lies in its ability to combine the design flexibility of plastic injection molding and the nearly unlimited choice of material offered by powder metallurgy, making it possible to combine multiple parts into a single one [1]. Furthermore, PIM overcomes the dimensional and productivity limits of isostatic pressing and slip casting, the defects and tolerance limitations of investment casting, the mechanical strength of die-cast parts, and the shape limitation of traditional powder compacts [2].

Typical parts produced by PIM range from milligrams to a few hundred grams [3]. It is important to mention that this technology is recommended for the mass production of small complex parts, due to the high capital investment in making high precision moulds with complex geometry. Therefore it is suitable for parts to be used in automotive or aerospace systems, and electronic devices, to mention a few.

The PIM process presents several variations that are used in the industry today. Invariably, it consists of four steps: (1) mixing metal or ceramic powder and a polymeric binder, (2) injecting this mixture into mold, (3) debinding to remove polymer from mixture and (4) sintering to bring together the otherwise loose powder [4]. All these steps are schematically shown in Figure 1.

One of the interesting applications of PIM is in the manufacturing of parts for moving vehicles because PIM can be used to combine multiple parts into a single part with complex geometry, and parts are lighter since porosity remains in the final part, at the same time the fatigue resistance increases. Fatigue resistance increases since the small pores act as crack stoppers, therefore preventing crack propagation and failure of a part loaded cyclically. Thus, it can be said that PIM can help reduce the weight of moving vehicles, which is a major concern from the environmental and economic point of view.

Ceramic injection molding has been around since 1940's [5] and metal injection molding since the 1970's [6], in fact, some of the first applications of metal injection molding was for the aerospace industry. Some examples include flat screw seals and rocket burning system, which were produced at the end of the 1970's [7]. In the automotive industry metal injection moulding is being utilized for manufacturing lock caps, lock shafts, cable seals used in sunroofs, soft magnetic sensor housing parts, cams in electrical systems of seats, bonnet lock fixing bearings, turbocharger vanes, rocker arms in engines, rollers and adjustment rings. One notable example of a automotive part produced by ceramic injection molding is a turbine wheel [1].

Even though the technology has been around for quite some time, there is still room for improving the process and materials utilized in PIM. One of the main limitations of current PIM is the long debinding time, which has been partially solved by introducing binder systems that undergo catalytic sublimation (polyoxymethylene-based binders); thus the molded part is debound in the solid state without melting or dissolving the polymeric binder in a much shorter time. However, current catalytic binders have the main problem of high viscosity. In this study, two ways to decrease the viscosity of PIM feedstock materials with polyoxymethylene were investigated. The first way was to reduce the average molecular weight of the binder [8] and the second one to select a polydisperse particle size distribution with high maximum packing fraction.

2. Materials and Methods

2.1. POM-based binders

Polyoxymethylene (POM) also known as polyacetal and less commonly as polyformaldehyde is a type of semicrystalline thermoplastic, linear polymer produced by chain growth polymerization. POM is generally synthesized as a homopolymer or copolymer. For this investigation, POM copolymers of different average molecular weight (Mw) were synthesized at BASF (Ludwigshafen, Germany). The nomenclature and average molecular weight of all the POM materials used in this study is shown in Table 1. Molecular weights were measured by the supplier using gel permeation chromatography.
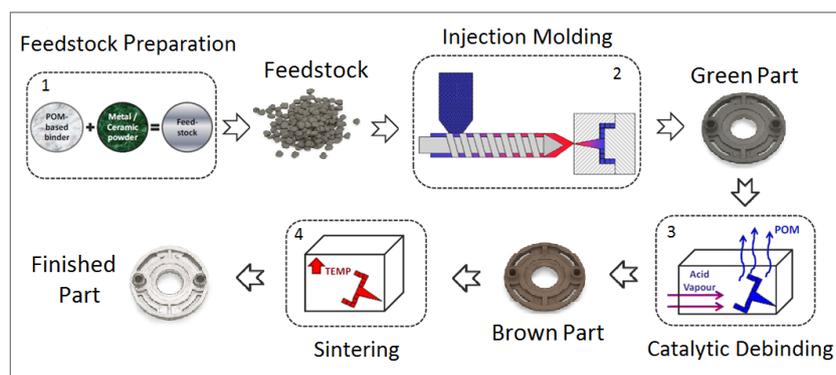
Figure 1. Flow chart illustrating the main stages of PIM process.

Table 1. Average molecular weight of POM copolymers.

Material ID	Average Molecular Weight, Mw, [g/mol]	Description
MW010	10240	Virgin POM copolymer, laboratory scale synthesis
MW024	24410	
MW052	52750	
MW081	81100	
MW092	92360	Virgin POM copolymer, industrial scale synthesis
MW109	109000	
MW129	129300	
MW204	204400	

2.2. Metal powders and feedstock materials

Stainless steel 316 is a chromium-nickel-molybdenum austenitic stainless steel developed to provide corrosion resistance to traditional alloys such as 304. The grade 316LW has a lower content of carbon (0.03 wt%) compared to 316 (0.08 wt%), which provides added corrosion resistance. Therefore 316LW can be used in the manufacturing of parts to be used by the maritime, automotive and aerospace industry.

For this investigation stainless steel 316LW powder with a broad particle size distribution (PSD) was supplied by BASF (Ludwigshafen, Germany). The powder was produced via gas atomization. The supplier separated the metal particles of the commercial product in five different fractions, each of these with different PSD and different average particle size (Figure 2). Particle size distribution was measured by laser scattering at BASF.

The matrix used in the suspension was polyoxymethylene (POM) copolymer with an average molecular weight of approximately 24400 g/mol. The POM copolymer was also produced by BASF (Ludwigshafen, Germany).

Feedstock materials with different solid content (20 to 45 vol %) were produced by extruding the steel powder with the POM copolymer at 190 °C. Mixing of copolymers was done in a twin-screw extruder (Haake Polylab, Thermo Scientific, Germany). In order to ensure proper mixing, extrudate was pelletized after the first extrusion and extruded once again. Number of extrusions was limited to two in order to prevent mechanical and thermal degradation of POM copolymers. The temperature profile and screw rotational speed of the extruder were set in such a way as to ensure that the POM melt did not exceed 190 °C to prevent thermal degradation. In order to determine the final content of solids (φ), the binder was removed by heating up the feedstock material up to 300 °C for two hours, and the remaining mass was used to estimate (φ).

2.3. Shear viscosity measurements

Viscosity measurements in oscillatory mode were performed in a MARS-II rotational rheometer (Thermo Scientific, Germany). Viscosity tests were performed at 190 °C, which is within the rage of temperatures at which POM is generally processed (180 to 230 °C). A truncated cone-plate measuring-geometry with a 20 mm diameter and angle of 1° was used. Two frequency sweeps were performed in each measurement; the first one increasing from 0.01 Hz (0.0628 rad/s) to 100 Hz (628.32 rad/s), and the second one decreasing from 100 to 0.01 Hz. All viscosity measurements at a given temperature were performed six times per material. In this study, viscosity results are presented as the magnitude of the complex viscosity ($|\eta^*|$), which is related to the constant rotational viscosity (η) through the Cox-Merz rule [9].

Constant rotational shear viscosity of the resulting feedstock materials was measured in a rotational rheometer fitted with parallel plates of 20 mm diameter at 180 °C (MARS II, Thermo-Scientific, Germany). All measurements were performed in triplicate. Shear rate was varied from 0.1 to 100 s^{-1}.
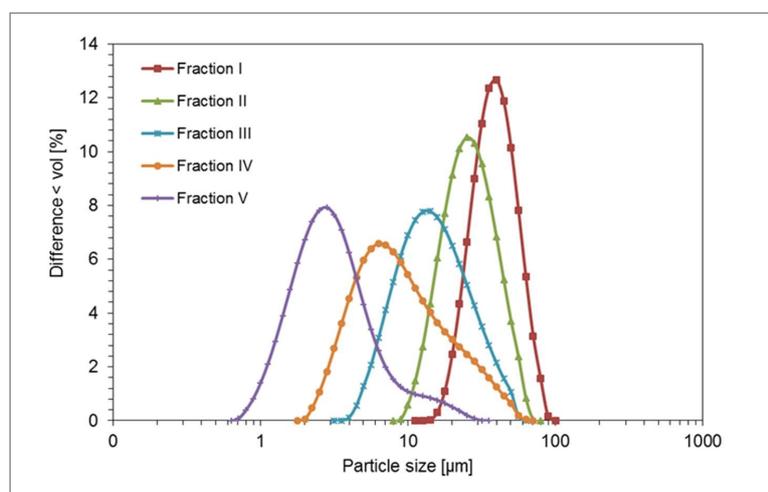
Figure 2. Differential volumetric particle size distribution for steel powder fractions.

3. Results and Discussion

3.1. Shear viscosity measurements of binder

The magnitude of the complex viscosity as a function of angular frequency for all POM copolymers was measured at 190 °C. From the measured complex viscosity data ($|\eta^*|$) the Newtonian viscosity (η_0) was estimated from the plateau at frequencies below 10 rad/s. The results are presented as a function of the average molecular weight in Figure 3. As with other polymers, POM copolymers show a rapid decrease in viscosity as the average molecular weight decreases following a power function as proposed by Fox and Flory [10]. The propose equation for POM copolymers is shown inside Figure 3.
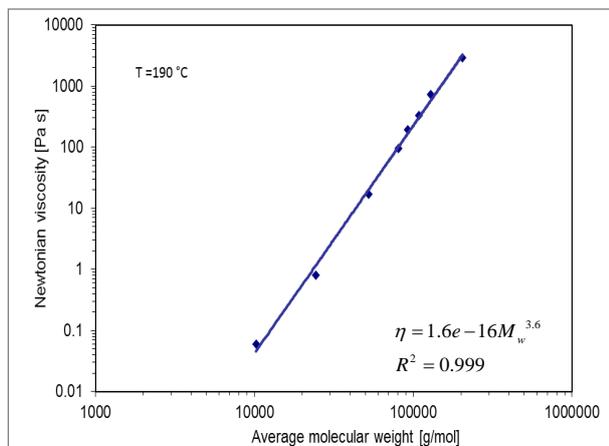
Figure 3. Newtonian viscosity of POM copolymers at 190 °C; the solid line represents a power law fit to the experimental data.

With respect to the selection of an appropriate binder for PIM, one could choose between the first three molecular weights (MW010, MW024 and MW052), which are all around the recommended viscosity of 10 Pa s [5]. For the purpose of this investigation we selected the middle one (MW024) for preparing feedstock materials.

3.2. Shear viscosity measurements of feedstock

Viscosity results for feedstock materials as a function of volumetric solid content (φ) are shown in Figure 4. Please notice that relative viscosity (η_r) is used in the vertical axis, which is the ratio of the viscosity of the suspension over the viscosity of the polymeric matrix, both viscosities measured under the same shear rate (100 s^{-1}) and temperature (180 °C). As expected, as φ increases so does the relative viscosity η_r, but the increase is significantly different depending on the fraction used in the feedstock material, since each fraction has different particle size distribution (Figure 2). Using Fraction V, the highest φ was achieved without increasing much η_r.

Five models were selected from the literature (Table 2) that predict the viscosity of concentrated macroscopic suspensions just by knowing the particle loading (φ) and the maximum packing fraction (φ_m), which represents the particle loading at which the viscosity goes to infinity. Other models were available in the literature, but they were ignored since they depend on other empirical parameters or are not applicable for concentrated suspensions.
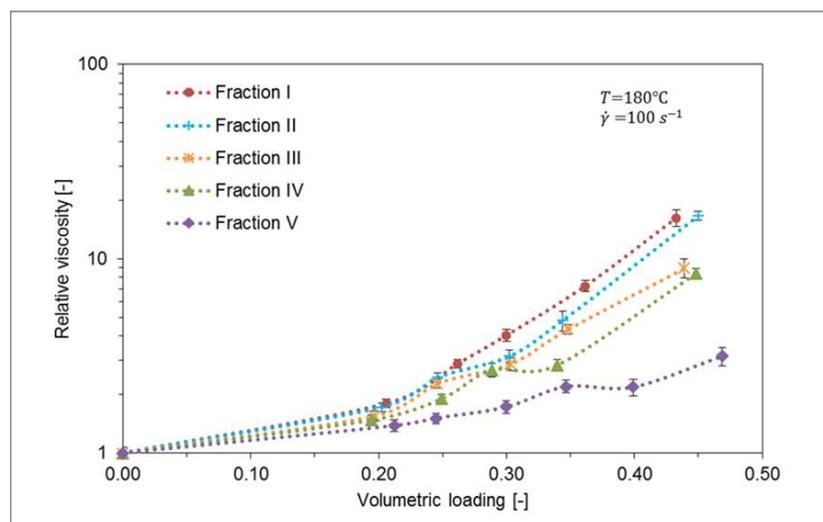
Figure 4. Relative shear viscosity η_r at 100 s^{-1} and 180 °C as a function of particle load φ for prepared feedstock materials containing powder fractions I-V.

Table 2. Models to predict the relative viscosity of concentrated suspensions.

Authors	Year	Equation
Frankel & Acrivos [11]	1967	$n_r = \dfrac{9}{8}\dfrac{(\phi/\phi_m)^{1/3}}{1-(\phi/\phi_m)^{1/3}}$
Chong et al. [12]	1971	$n_r = \left[1+0.75\left(\dfrac{\phi/\phi_m}{1-\phi/\phi_m}\right)\right]^2$
Quemada [13]	1976	$n_r = \left(1-\phi/\phi_m\right)^{-2}$
Zarraga et al. [14]	2000	$n_r = e^{-2.34\phi}\left(1-\phi/\phi_m\right)^{-3}$
Mendoza & Santamaria-Holek [15]	2008	$n_r = \left(1-\dfrac{\phi}{1-c\phi}\right)^{-2.5}$ $\quad c = \dfrac{1-\phi_m}{\phi_m}$

Data shown in Figure 4 was fitted with the five models selected and (φ_m) for each powder fraction was estimated. The coefficient of determination (R^2) was used to determine which model fits the data better overall. The results for the best (Zarraga et al) model are shown in Table 3.

Using the model proposed by Zarraga et al, one can estimate the maximum loading needed to achieve a viscosity close to 1000 Pa s at the selected conditions for each fraction. Table 3 shows that Fraction V has the highest (φ_m) and therefore it can be loaded the most without increasing the viscosity beyond 1000 Pa s, approximately to up to 83 %vol.

4. Conclusions

Reduction of viscosity of PIM feedstock is very important, since it will facilitate the injection molding of parts with complex geometry. In this study it was observed that viscosity of POM copolymers increases with average molecular weight following a power law relationship. With these results on hand, we recommend to use a POM-based binder with a molecular weight around 24400 g/mol. Feedstock materials were prepared with the recommended binder at different powder loadings. It was observed that viscosity of feedstock materials for PIM can be further reduced by selecting the appropriate particle size distribution. In this study it was shown that using powder with a wide particle size distribution leads to higher maximum packing fraction that can reduce significantly the viscosity of feedstock materials. It was also observed that from the simple models available in the literature the model of Zarraga et al [14] best fits our experimental data. Using this model the maximum loading for the feedstock to reach a viscosity of approximately 1000 Pa s using the powder with the highest maximum packing fraction is approximately 83 %vol.

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Table 3. Maximum packing fraction (φ_m) and coefficient of determination (R2) obtained by fitting at 100 s^{-1} and 180 °C by Zarraga et al model [11].

Powder Fraction	Maximum packing fraction, φ_m	Coefficient of determination, R^2	Maximum loading φ before η_r reaching 1000 Pa s
I	0.602	0.997	0.554
II	0.622	0.998	0.573
III	0.666	0.996	0.615
IV	0.689	0.985	0.637
V	0.889	0.971	0.832

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