

## DRY MIXING OF FEEDSTOCK FOR INJECTION MOULDING OF 316L STAINLESS STEEL POWDER

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**Abstract.** An injection moulding binder, which is composed of poly ethylene glycols (PEGs) of various molecular weights and poly methylmethacrylate (PMMA), has been developed for injection of metal powders. Feedstocks have been prepared using a wet process with PMMA introduced in the form of an emulsion with particles of 0.1 to 0.2  $\mu\text{m}$  in size. To eliminate the drying process, an attempt has been made to prepare the feedstock using a dry mixing process using 316L stainless steel powder as a model material. For this, the PMMA used was either a commercial powder, Elvacite, with a particle size of up to 100  $\mu\text{m}$  or a powder obtained by drying the emulsion and grinding the resultant cake, followed by classification using 53  $\mu\text{m}$  sieves. The feedstock constituents were mixed into a sigma blade mixer for 2 hours at mixing temperature of 120°C. The feedstock was then hot extruded using piston injection moulding machine prior to being moulded. It was found in all cases that PMMA was poorly dispersed. This resulted in an increased tendency for the moulded bars to swell on being leached and the sintered bars to have a substantial residual porosity. This study suggests that by using small particles of PMMA powder, an acceptable product could be obtained.

*Keywords:* Dry mixing, metal injection moulding, sintering, feedstock, 316L stainless steel powder

**Abstrak.** Bahan pengikat pengacuan yang terdiri daripada polietilena glikol (PEG) dan polimetil metakrilate (PMMA) telah dihasilkan untuk proses pengacuan suntikan logam. Penyediaan bahan suapan adalah secara kaedah basah di mana PMMA adalah dalam bentuk emulsi dengan saiz serbuk antara 0.1  $\mu\text{m}$  hingga 0.2  $\mu\text{m}$ . Oleh itu, untuk menghindarkan proses pengeringan, satu kaedah baru penyediaan bahan suapan dalam bentuk percampuran kering telah dilakukan. Untuk ini, PMMA yang digunakan adalah terdiri daripada serbuk PMMA komersial berjenama Elvacite yang mempunyai saiz serbuk 100  $\mu\text{m}$  dan juga serbuk PMMA yang didapati daripada proses pengeringan emulsi yang dikisar dan diayak hingga ke saiz serbuk 53  $\mu\text{m}$ . Bahan suapan tersebut telah dicampur ke dalam mesin pencampur bilah sigma selama dua jam pada suhu 120°C. Bahan suapan kemudian disemperit panas sebelum dilakukan proses pengacuan. Keputusan uji kaji menunjukkan dalam semua kes, didapati PMMA telah menyerak secara tidak sekata. Ini seterusnya meningkatkan lagi bahan teracu untuk mengampul semasa proses pengurusan dan menyebabkan bahan tersinter mempunyai banyak keliangan. Walau bagaimanapun, adalah dirumuskan bahawa dengan menggunakan serbuk PMMA yang bersaiz lebih kecil, produk yang lebih baik akan diperolehi.

*Kata kunci:* Percampuran kering, pengacuan suntikan logam, pingsinteran, bahan suapan, serbuk keluli tahan karat 316L

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## 1.0 INTRODUCTION

The emphasis on miniaturation, design flexibility, and low cost has stretch the capabilities of conventional manufacturing route to the limit. An approach to meet such challenge has been the development of metal injection moulding (MIM). This process technology is an elegant blend of plastic injection moulding, based on the use of fine powder particles mixed with waxes and/or thermoplastic polymers to form a feedstock that can be moulded. The granulated feedstock is then given a shape using an injection moulding machine. After shaping, the polymer binder must be removed from the moulded part without significantly disturbing the powder particles. Then, the powder is sintered at high temperatures, often to near theoretical densities [1-3].

A new binder system has been developed which employs a mixture of polyethylene glycols (PEGs) with different molecular weights, and poly methyl methacrylate (PMMA) introduced in the form of an emulsion [4-7]. This binder system has the advantage that the major part, the PEGs, can be removed from the moulding by water leaching. This allows rapid removal of the residual binder, the PMMA, by thermal pyrolysis. Thermal degradation of the binder is the normal way in which debinding is carried out and for most binders, this involves a very long heat treatment, which usually takes several days to complete.

The original method of feedstock preparation involves mixing the powder with water based emulsion of PMMA and dissolving PEGs in water in the form of paste [8]. This formation is dried for 24 hours and given a short hot mixing which, with small laboratory scale batches, is carried out with a pallet knife. An industrial user has stated that the rapid removal of PEG/PMMA binder is a worthwhile advantage but the use of wet mixing approach to form the feedstock, which requires a drying step, is not ideal when industrial scale batches have to be produced.

Thus, it was decided to try to develop a method of feedstock production, which eliminated the drying process. To be successful, a dry mixing method for feedstock preparation must disperse the PMMA as well as is achieved by wet mixing, to obtain high sintered density. This is required as several properties including strength, improve with density. In this study, the feedstock was prepared by melting all the binder constituents and lubricant together and then hot mixing them with metal powder using a sigma blade mixer.

## 2.0 MATERIALS AND METHODS

The powder used in the present study was obtained from Manganese Bronze Components as a classified gas atomised 316L stainless steel powder. Two types of stainless steel powders were used, namely coarse and fine powders. The chemical compositions of the powders given by the manufacturer are given in Table 1.

The particle size distribution was determined using a Coulter LS 130 laser particle size analyser and is given in Table 2. It shows that the powders had a median particle

**Table 1** The chemical compositions of the 316L stainless steel powder

Type	Elements, %							
	C	Si	Mn	P	S	Cr	Ni	Mo
coarse	0.090	0.32	0.80	0.041	0.016	16.40	12.40	2.31
fine	0.017	0.69	1.32	0.030	0.002	17.19	13.82	2.77

**Table 2** The cumulative particle size distribution of stainless steel powder

Type	% <	10	25	50	75	90
coarse	mm	9.8	13.9	24.1	32.8	40.0
fine	mm	4.1	6.8	12.6	20.4	30.3

size of around 24 (coarse) and 13 (fine)  $\mu\text{m}$ . It can be seen that the powder had a relatively wide particle size distribution which is desirable for efficient particle packing. Scanning electron microscopy was used to determine the particles morphology and it shows that all particles were approximately spherical.

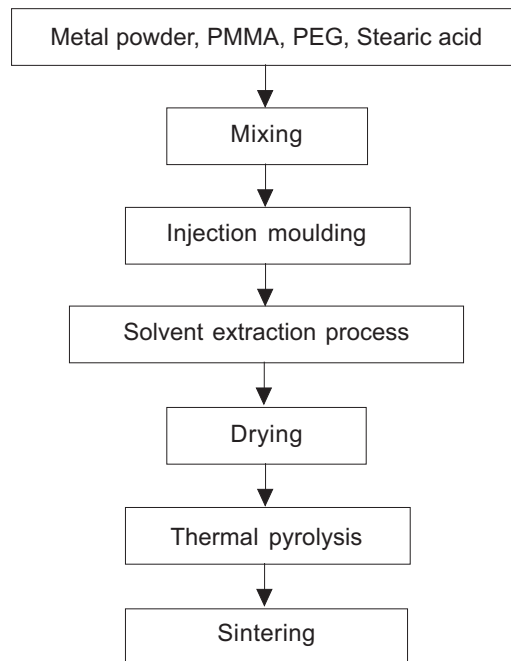
The two component binders consist of a major fraction (75 – 90%) of PEGs, in this case with different molecular weights of 600 (10 wt%), 1000 (10 wt%), and 1500 (80 wt%). Two forms of PMMA were employed in this study. One was obtained from a PMMA emulsion, by grinding the cake formed on drying the emulsion at 60°C and was specially prepared by Scott Bader Co Ltd, Wellingborough, UK. The other was a PMMA powder, ICI Elvacite, which is composed of spherical particles with diameters up to 100  $\mu\text{m}$ .

The feedstock prepared in this study contained of 67.5 vol.% of stainless steel powder. The composition of the binder used in this study was 2 wt.% stearic acid, 8.3 wt.% PEG<sub>600</sub>, 8.3 wt.% PEG<sub>1000</sub>, 66.4 wt.% PEG<sub>1500</sub>, and 15 wt.% PMMA, either Elvacite or powder from dry emulsion. To prepare the feedstock, initially, PEGs, PMMA and stearic acid were heated in the Z blade mixer. Metal powder was gradually introduced into the melt. The powder-binder mixture was mixed for 2 hours at the temperature of 120°C.

Injection moulding was performed using a simple, vertically aligned and pneumatically operated plunger machine. A barrel equipped with a thermocouple near the nozzle, was heated up by a jacket heater. A feedstock was fed into the barrel, extruded to improve homogeneity, and then chopped into small pieces about 5 mm in length. Chopped pieces were fed into the barrel and then injected through nozzle into the mould cavity. The pressure was held for about 3 seconds for proper mould filling. Moulded specimens were allowed to cooled down until they were firm enough to be removed from the mould. A simple bar specimens of 55 mm length, having a square cross section of 5 × 5 mm were moulded at 46 MPa. The green (as-moulded)

strengths of the mouldings were determined using a 3-point bend test. The diameter of the support rollers was 3.15 mm and the span length was 25.4 mm. The loads at fracture were recorded.

Debinding was performed in two steps, i.e. water leaching to remove the PEGs and thermal pyrolysis to remove PMMA. Green specimens were immersed in distilled water, held at the temperature of 60°C for 240 minutes. The specimens were supported on glass beads so that the binder would be leached at a similar rate from the top to the bottom surfaces. Subsequently, the partly debound specimens were dried at the temperature of about 50°C for about 1 hour to evaporate water from the specimens. For thermal pyrolysis, the leached specimens were put into an alumina tube and then the surrounding space was filled with alumina powder to provide support under argon atmosphere. The thermal pyrolysis cycle consists of heating at a rate of 3°C/min to 400°C and soaking for 30 minutes. The sintering process was performed in the same furnace as that used for residual binder removal. Heating rate of 5°C/min is used from 400°C to the sintering temperature of 1360°C. Finally, the bars were soaked for 240 minutes and furnace cooled.

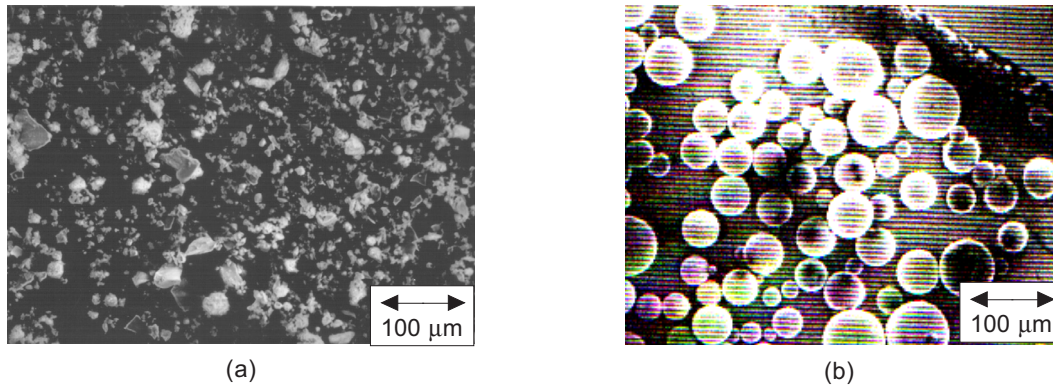


**Figure 1** Flow diagram of the overall process

### 3.0 RESULTS AND DISCUSSION

#### 3.1 Feedstock Preparation

The binder mixture became more and more viscous during melting as the PMMA dissolved into the PEGs. This occurred for both types of PMMA, i.e. the Elvacite and the powder produced by grinding the cake formed on dried Scott Bader emulsion. The particle morphology of both PMMA powders are shown in Figures 2a and 2b. The process of mixing was difficult if metal powder was added when the binder was highly viscous. Therefore, the metal powder was added to the molten binder before it became too viscous to be mixed in the Z-blade mixer. In this state, neither type of PMMA had been completely dissolved in the PEGs.



**Figure 2** Scanning electron micrographs of the PMMA powder used in dry feedstock preparation. (a) Dry PMMA emulsion after being sieved 53 mm, and (b) ICI Elvacite PMMA powder

#### 3.2 Moulding Behaviour and As-Moulded Strength

The feedstocks prepared in this study contained 67.5 vol.% of coarse or fine stainless steel powder. The compositions of the binder used in this study were 2 wt.% stearic acid, 8.3 wt.% PEG<sub>600</sub>, 8.3 wt.% PEG<sub>1000</sub>, 66.4 wt.% PEG<sub>1500</sub>, and 15 wt.% PMMA either Elvacite or powder from a dried emulsion. The moulding pressure was kept constant at 46 MPa but the temperature was changed until satisfactory moulding conditions were obtained. The temperatures employed are given in Table 3.

**Table 3** The moulding temperatures of feedstocks made with coarse and fine powders using a dry preparation method

Type of PMMA	Moulding temperature, °C	
	coarse powder	fine powder
ICI Elvacite powder	120 – 125°C	125 – 127°C
Dried PMMA emulsion	125 – 130°C	130 – 135°C

The feedstock was made with coarse metal powder, prepared with ICI Elvacite PMMA powder, and moulded at temperatures around 120 – 125°C. For the fine powder, the feedstock required a slightly higher moulding temperature of between 125 and 127°C. These moulding temperatures are lower than that is usually required for moulding a feedstock prepared using a wet mixing approach [ 5, 7]. This could be the reason that PMMA was not as well dispersed into the PEGs, thereby resulting in a lower viscosity. The moulded bars had to be left for 2 to 3 minutes in the mould before they could be removed without damage, because of their softness, which is relatively shorter compared with the feedstock prepared using the wet mixing approach.

For the feedstocks prepared using the ground, dried emulsion, the mould cavity was not successfully filled using the same moulding condition. The temperature was increased to about 130°C for the feedstock made with coarse powder and to 135°C for the one made with fine powder. This suggested that the finer PMMA was more effectively dispersed, which resulted in increased viscosity. The mouldings were soft and flexible for the extended time. Thus, distortion occurred during removal from the mould unless a setting time of about 3-4 minutes was allowed. This setting time is closer to the time required for feedstocks prepared by the wet mixing approach [5, 7].

The reasons for the increase in the setting time needed upon the introduction of stearic acid and the reductions encountered in these dry mixing approaches are not understood at the present time.

The results of the as-moulded and as-leached strength measurements are shown in Table 4 for the coarse and fine powders, respectively. The feedstocks made with the dried emulsion powder gave slightly higher values for both metal powders. This is consistent with the view that the dispersion of PMMA was better when the ground PMMA powder obtained from the dried emulsion was used. For example, comparing the mouldings made with Elvacite powder and the powder derived from dried emulsion for the fine metal powder, the strengths in the as-moulded state were 7.8 MPa and 8.5 MPa, respectively. Surprisingly, the as-moulded strengths for the bars made by dry mixing routes were slightly higher than those made using wet mixing, which were 5.5 MPa and 7.1 MPa for the coarse and fine powders, respectively [8].

However, the as-leached strengths of the bars made by wet mixing were stronger than those made by the dry mixing routes. For the bars made with fine powder by wet mixing route were 1.4 MPa, compared with 0.8 MPa and 1.1 MPa for the materials made with Elvacite, and dried emulsion powder respectively. These results would be expected because of poorer dispersion of PMMA in the dry mixing route, as the as-leached strength arises from PMMA binding the powder particles together.

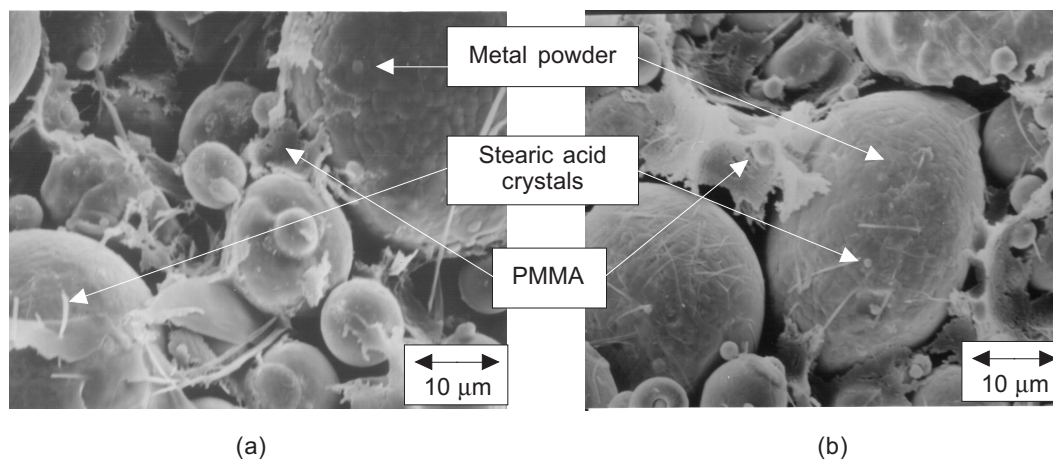
**Table 4** The as-moulded and as-leached strength of the specimens (mean values and standard deviations)

Type of PMMA	As-moulded strength, MPa		As-leached strength, MPa	
	fine	coarse	fine	coarse
ICI Elvacite	7.8 ± 0.4	7.5 ± 0.3	0.8 ± 0.1	0.6 ± 0.1
PMMA dry emulsion	8.5 ± 0.5	7.5 ± 0.2	1.1 ± 0.2	0.5 ± 0.1

### 3.3 Water Leaching and Thermal Pyrolysis

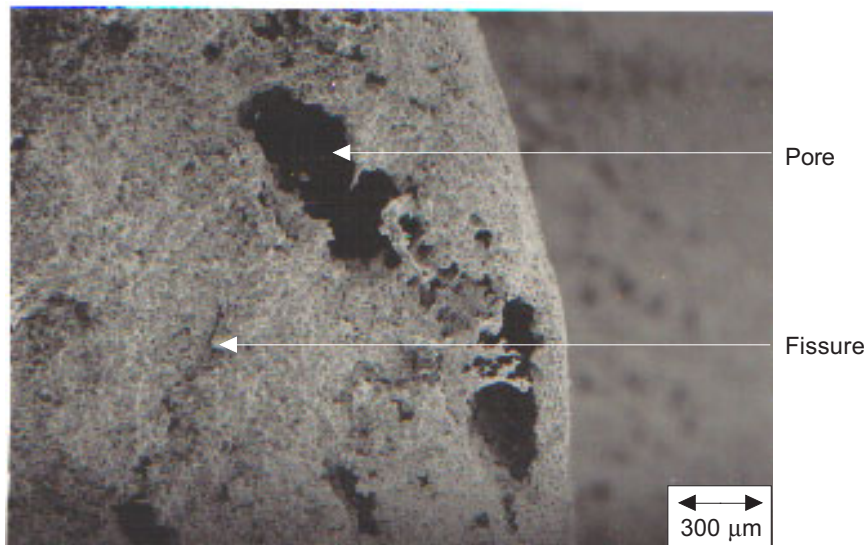
Leaching for 4 hours in warm water (60°C) was found to be sufficient to totally remove PEGs from the mouldings made with 67.5 vol.% loading and to open-up pore channels for subsequent thermal pyrolysis. However, it led to significant changes in the dimensions and swelling, whereas leaching at a lower temperature provided better shape retention. Using the wet mixing process, the dimensional changes were usually a fraction of 1 to 1.5% [5] but using the dry mixing approach, the changes were 3 to 5%. This can be expected if the PMMA is poorly distributed and dispersed, as less PMMA will, in effect, be available to hold the metal particles together.

Figures 3a and 3b show scanning electron micrographs of mouldings made using the PMMA obtained from the emulsion and Elvacite PMMA powder after leaching for 4 hours. The network of ligaments of PMMA that hold the particles together has a similar morphology in two cases. Crystals of stearic acid can be seen on the surfaces of the metal particles. Figure 4 shows large fissures and pores in leached mouldings made with dried PMMA emulsion powder resulting from poorly distributed PMMA.



**Figure 3** Scanning electron micrographs of mouldings made with the 316L stainless steel powder with binder prepared with (a) PMMA obtained from the emulsion, and (b) Elvacite PMMA powder after leaching for 4 hours at 60°C

Thermal pyrolysis to remove the remaining PMMA was carried out in a tube furnace with the presence of flowing argon to prevent oxidation. A heating rate of  $3^{\circ}\text{C}/\text{min}$  up to  $400^{\circ}\text{C}$  was used and the mouldings were held at this temperature for 30 minutes to remove all the PMMA. Sintering was performed in the same tube furnace using either argon or  $95\%\text{N}_2/5\%\text{H}_2$ . Heating rate of  $5^{\circ}\text{C}/\text{min}$  from  $400^{\circ}\text{C}$  to  $1360^{\circ}\text{C}$  was used. The mouldings were soaked at the upper temperature for 4 hours.



**Figure 4** Scanning electron micrographs showing large fissures and pores in leached mouldings made with dry PMMA emulsion resulting from the poorly distributed PMMA

### 3.4 Microstructure of the Sintered Specimens

Table 5 shows the sample densities attained by sintering in different atmospheres for coarse and fine powders. It is clearly shown that mouldings made with fine powder exhibit higher density than those made with coarse powder. For both dry PMMA

**Table 5** The sintered density of the test specimens at different sintering atmospheres (% of theoretical maximum value)

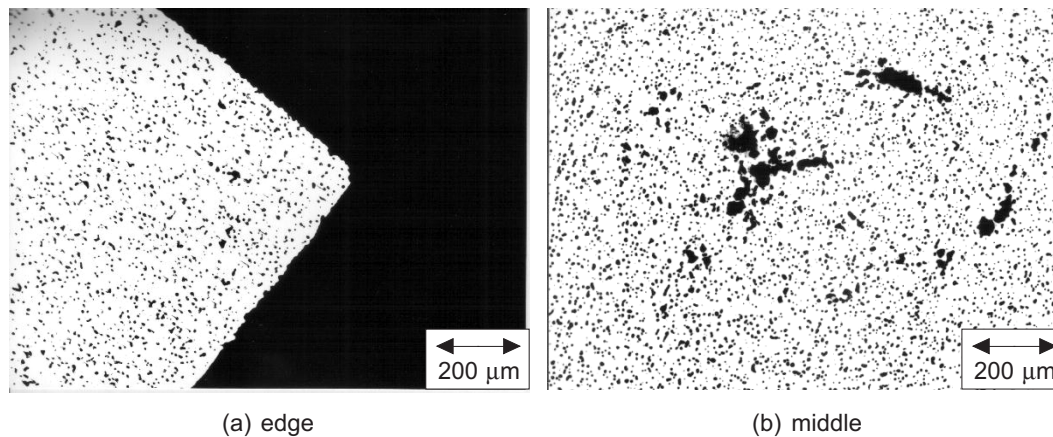
PMMA type	Metal powder and atmospheres			
	fine powder		coarse powder	
	argon	$95\%\text{N}_2/5\%\text{H}_2$	argon	$95\%\text{N}_2/5\%\text{H}_2$
ICI Elvacite	96.8%	97.2%	94.7%	94.9%
Dried emulsion	97.1%	97.2%	94.1%	94.8%



emulsion and Elvacite PMMA, the highest sintered density that could be attained using coarse metal powder was about 95% of the theoretical maximum value. As expected, the mouldings made with finer powder exhibited higher sintered densities, close to 97% for both PMMA types.

Comparing the samples made using the wet mixing approach with the same sintering conditions, it appears that dry feedstock preparation gave lower sintered densities, which were about 1.5% lower for both powders. This was presumably caused by the comparatively poorer distribution of PMMA in the mixtures, although this should have been improved and the sintered density increase for a longer mixing times.

Figures 5a and 5b show the microstructures of sintered samples made with the coarse stainless steel powder using Elvacite PMMA powder. Numerous pores were observed measuring around 15 to 30  $\mu\text{m}$  in size.



**Figure 5** Optical micrographs of a sintered specimen made with coarse powder from a feedstock prepared using dry mixing route with Elvacite PMMA powder after sintering in argon at 1360° C for 240 minutes

#### 4.0 CONCLUSION

Failure to disperse PMMA on a sufficiently fine scale of size has been shown to make mouldings prone to swelling during leaching and limit the density to which they can be sintered. Using the dry mixing method, the highest sintered attained was close to 97% of theoretical density. These results would be expected because of the poorer dispersion of PMMA in the dry mixing route. However, the densities achieved are sufficiently close to the best values achieved by wet mixing to suggest that a dry mixing approach could be successful if mixing were to be carried at longer time and smaller PMMA particles were used.

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