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Low Pressure Powder Injection Moulding of Stainless Steel Powders

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Abstract. Low-pressure powder injection moulding was used to obtain AISI 316L stainless steel parts. A rheological study was undertaken using gas-atomised powders and binders. The binders used were based on carnauba wax, paraffin, low density polyethylene and microcrystalline wax. The metal powders were characterised in terms of morphology, particle size distribution and specific surface area. These results were correlated to the rheological behaviour. The mixture was injected in the shape of square bar specimens to evaluate the performance of the injection process in the green state, and after sintering. The parameters such as injection pressure, viscosity and temperature were analysed for process optimisation. The binders were thermally removed in low vacuum with the assistance of alumina powders. Debinding and sintering were performed in a single step. This procedure shortened considerably the debinding and sintering time.

Introduction

The metal powder injection process has been used since 1920 and consists of mixing metal powders with organic binders, debinding and sintering. The technique follows the same principles of plastic injection moulding and has many technological advantages, including the production of a large number of small and intricate parts. The process can be divided into high pressure, i.e., above 0.7 MPa and low-pressure. The high-pressure method has the advantage of producing large parts and, the disadvantage of high die costs. In low pressure injection moulding, obtaining parts larger than 10 cm can be critical, depending on its mass and geometry. The low pressure method has some advantages such as low die costs and low equipment service. The injection moulding process allows the use of metallic, ceramic, composite and biomaterial powders for component production [1,2].

The demand for small parts with complex geometries is concentrated mainly in two markets, electro-electronics and high-precision mechanics. The current market is favourable for engineering plastics such as nylon, due to its low cost and versatility. With the appearance of metal injection moulding (MIM), metals can claim their market share, not only in the electro-electronics and high-precision mechanics areas, but also in the aeronautics and surgical implants areas. These markets require low cost production of highly elaborate and accurate parts [3].

The low-pressure metal injection moulding process produces parts with reduced internal stresses. This results in components with few defects after binder removal. Conversely, the low-pressure method limits the choice of binders, due to low viscosity restrictions. The binders indicated are wax and low molecular weight polymers, and a maximum volume of solids of 65 % [1,4]. In this

case, the success of the method depends on rheological awareness (viscosity, metal powder shear rate during injection in the mould and injection temperature). Further, it is also mandatory to know well, the powder characteristics such as particle size distribution, specific surface area, degree of moisture, mean size of particles and morphology. These items determine the success or failure of the process.

Experimental

Very fine gas atomised powders (below 25 μm) of stainless steel AISI 316L were used. The particle size distribution was characterised by laser diffraction. The morphology of the powders was examined by scanning electron microscopy - SEM. The moisture content of the powders was measured after drying the powders at 120 °C for 4 hours. The mean diameter of the particles was determined with a Fisher sub-sieve sizer, which gave sound results for spherical particles. The Fisher sub-sieve size analysis is applicable to spherical particles ranging from 0.2 to 50 μm in size and is based on the flow rate of gas passing through the particles. The variation of gas flow rate with pressure, is used for mean particle size determination using equation (1):

$$S = 6/\rho d \qquad (1)$$

where: ρ is the theoretical density of the metal, d is the mean particle size and S is the specific surface area. The specific surface area was also measured by the BET method and its results were compared with those obtained with equation (1).

The powder mixture can be considered to be a modified polymeric system. The metal powder with binder mixture was obtained under constant stirring at 145 °C. The homogeneity of the mixture was evaluated by fractography of samples and by rheological measurements carried out with a microcomputer controlled digital rheometer.

In the metal-polymer mixture, the amount of binder was 38.0 % by volume, and consisted of 5.5 % of low molecular weight polyethylene, 22.5 % of paraffin, 8.0 % of carnauba wax and 2.0 % of microcrystalline wax. The low-density polyethylene was used to confer green strength and to avoid specimen collapse during the transition stage between debinding and sintering. The paraffin reduced the metal-polymer system viscosity and corrected the rheological behaviour during injection. The carnauba wax was responsible for lubrication and acted as a demoulding agent. The microcrystalline wax, a polyolefine, had an effect similar to that of carnauba wax.

The specimens were injected at 0.4, 0.5 and 0.6 MPa pressure and at temperatures ranging from 135 to 155 °C. The results of the injection pressure variation were evaluated by means of green density measurements.

Thermal wick debinding was used to remove the binders from the compact, with the assistance of fine alumina powders that removed the binders by capillarity. The debinding was performed in a tubular furnace heated up to 600 °C for 4 h in 10^{-1} Torr vacuum. After binder removal, the bars were heated further to the sintering temperature of 1300 °C. This sintering temperature was established, based on prior dilatometric testing performed on pre-sintered samples.

Results and discussion

In powder characterisation, there were difficulties in assessing the particle size distribution. This was related to the use of an inadequate deflocculant and dispersant. The action of the dispersant is characterised by electric, stereo and electric-stereo effects. Bearing this in mind, an adquate dispersant for stainless steel was chosen from those commercially available. The dispersant that gave the best result was sodium polyacrylate, which showed the electric-stereo effects. The electric-stereo effect is susceptible to the powder atomisation variables, and consequently to changes in powder characteristics.

The particle size distribution evaluated by laser diffraction showed a bimodal distribution, as shown in Fig. 1. According to German [4], this can lead to a high packing density, which is a positive aspect for the injection moulding process at low pressure.

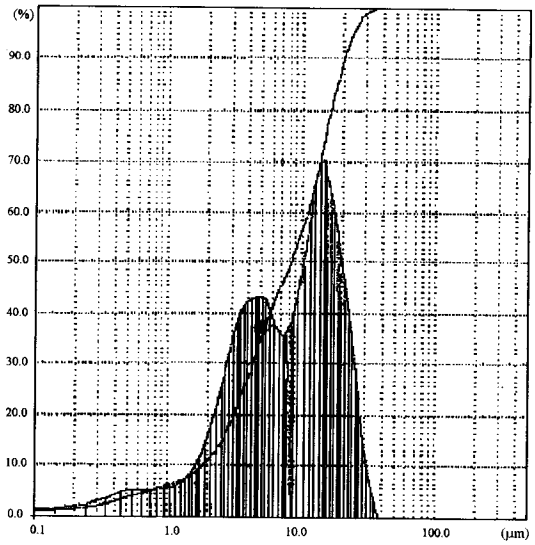

Fig. 1. Particle size distribution of the stainless steel powder obtained by laser diffraction.

The stainless steel powders were produced by gas atomisation. The powders were spherical, as shown in the secondary electron micrograph in Fig. 2. According to current literature [5,6], the main advantages of spherical morphology are reduced viscosity and good packing, which are favourable in terms of the injection moulding process, and avoid cavity formation in injected parts. The cavities (entrapped gas bubbles) can originate at the mixing stage (metal powders and polymers), due to the high temperatures, during which, binding agent evaporation could take place. This heterogeneity in the mixture can be detected via direct observations in the SEM and from discontinuities in the rheological measurements. These cavities lead to non-homogenous thermal distribution in the mixture, which upon sintering leads to fissure formation and warpage. The metal powder-polymer mixture can be seen in Fig. 3. Table 1 presents the density and viscosity data of the polymers that were used.

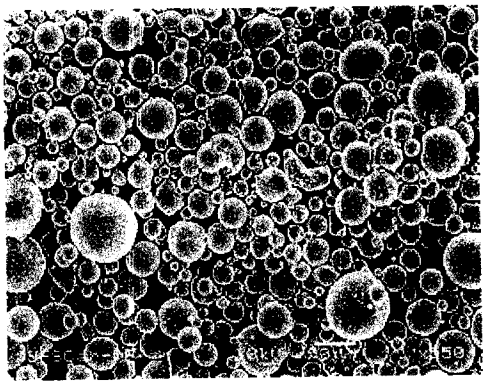

Fig. 2. Secondary electrons micrograph of the stainless steel powder used in this investigation, showing spherical morphology of the particles.

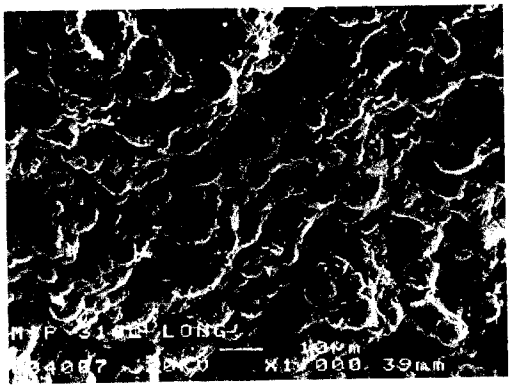

Fig. 3. Secondary electrons micrograph of green specimen fracture, showing particle wettability by the binder.

Table 1. Some physical properties of the binders used in this investigation.

Material	Density (g/cm^3)	Melting point. (°C)	Viscosity (Pa.s) at the ref. temp.	Ref. temp. (°C)
Polyethylene	0.92	113	0.79	130
Paraffin	0.90	62	0.009	101
Carnauba	0.99	86	0.0209	110
Microcrystalline wax	0.97	125	0.018	120

Once verified that the powder morphology was spherical, use of the Fisher sub-sieve size analyser for the mean particle size determination was decided upon, and consequently, the specific surface area calculated with equation (1). The mean particle diameter measured by the Fisher sub-sieve sizer was 8.24 μm, which is quite similar to the mean particle size calculated with equation (1), and the specific surface area obtained with the BET method. Thus, the Fisher sub-sieve sizer and the BET method gave a good correlation for spherical powders. The value for the specific surface area can be a good indicator of the powders reactivity. For spherical powders, which have low roughness,

a low surface energy is expected and consequently, a low degree of moisture. This was confirmed from moisture determinations (see Table 2). Regarding the remnant moisture, secondary chemical bonds can be generated, i.e. hydrogen bridges can be formed between the water adsorbed at the particle surface and the polymer binder. This can pose certain difficulties during debinding, contributing to crack and warpage formation. Ceramists have studied this aspect, and often reffered to it as water of crystallisation..

Table 2. Physical characteristics of the stainless steel AISI 316 L powders used.

Laser diffraction (μm)	Moisture (%)	BET specific surface area (m^2/g)	Fisher mean particle size (μm)	Particle morphology
d_{10} = 1.85 d_{50} = 8.25 d_{90} = 20.24	0.04	0.092	8.24	spherical

The best compromise between pressure and temperature for selected metal-polymer mixtures was obtained. At temperatures above 145 °C the binders evaporate too fast, but permit injection at low pressures, 0.5 MPa, where viscosity corrections are possible every time the green density of the part shift 5 % from the mean.

For wick debinding, the green part was mixed with fine alumina powder and put inside a vacuum retort at 10^{-1} Torr. The retort was heated up to 600 °C at 7 °C per minute. This debinding condition optimised the capillarity effect and consequently reduced the debinding time. Furthermore, the alumina powders favoured a homogeneous thermal distribution, which could be verified from reductions in sample warpage.

The rheological behaviour of the mixture was determined yo be pseudo-plastic, which is beneficial for the injection process[4,6]. Every time the rheological behaviour of the mixture departed from pseudo-plasticity, injection difficulties occurred. In such situations, the viscosity was corrected by paraffin addition, to make the rheological behaviour once again, pseudo-plastic.

Problems with mass heterogeneity or mould filing can be seen in Fig. 4. The irregular distribution of the polymer in the mixture can be detected by changes in rheological data, and corrected during the process.

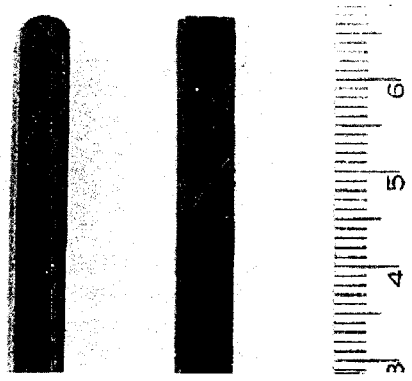

Fig. 4. Irregular mould filling indicating the jetting phenomenon.

Conclusions

The mixture of spherical powders and waxes were found to be efficient in lowering the interparticle friction, which is essential for the low-pressure injection process.

Particle size distribution measurement by laser diffraction can be accomplished by using sodium polyacrylate as the dispersing agent.

The pseudo-plastic rheological behaviour of the mixture can be controlled by simple density measurements.

It is recommended that before powders and binders are mixed, the metallic powders be heated to decrease the moisture content to around 10^{-2} %. This contributes to reduction in the number of secondary bonds between the metal particles and binders, affecting the most critical stage, debinding.

The use of wick powders and low vacuum were efficient for reducing the debinding time and for maintaining the sample shape during sintering.

Heterogeneity of the mixture that occurred during injection could be verified from the discontinuities in the rheological measurements.

The low pressure injection moulding process is practically scrap free, as the green parts can be reinjected after their rheological properties are corrected.

Low-pressure injection moulding has commercial potential for the production of a large number of small and intricate parts.

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