

Effect of powder particle shape and size distributions on the properties of low-viscosity iron-based feedstocks used in low-pressure powder injection moulding

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Low-pressure powder injection moulding (LPIM) is a cost-effective manufacturing technology used to fabricate complex-shaped parts with high mechanical properties at low- or high-volume production. This research work presents an experimental approach to investigate the debound/sintered properties of iron components produced by the LPIM process using iron-based powders exhibiting different particle shapes and size distributions. Four low-viscosity feedstocks were mixed and injected into a rectangular mould cavity before being thermally wick-debound and finally sintered using identical debinding and sintering cycles. This study confirms that both irregular and spherical iron powders can be shaped via the LPIM process. The solid loadings obtained with these two powder morphologies, varying from 58 to 62 vol. %, represent expected values. The trend in the density values obtained with irregular ($\sim 6.6 \text{ g/cm}^3$) and spherical ($\sim 7.5 \text{ g/cm}^3$) powders was validated by metallographic observations.

Keywords: Low-pressure powder injection moulding (LPIM); thermal-wick debinding; pre-sintering temperature; sintering; iron powder; irregular particles.

1 Introduction

Metal injection moulding (MIM) is a cost-effective near net-shape manufacturing technique that combines the advantages of powder metallurgy and plastic injection moulding technologies. In MIM, a molten polymeric binder is mixed with a metallic powder to produce a feedstock that is injected into a mould cavity, debound, and sintered to finally obtain dense parts [1–3]. Based on the binder system, the process is categorized as either high-pressure powder injection moulding (HPIM) or low-pressure powder injection moulding (LPIM). High-viscosity feedstocks (e.g., $> 100 \text{ Pa}\cdot\text{s}$) containing a backbone polymer generally require the conventional HPIM approach, which uses reciprocating screw machines to inject green parts at pressures ranging from 50 to 200 MPa [4–7]. By contrast, as it takes advantage of low-viscosity feedstocks (e.g., $< 20 \text{ Pa}\cdot\text{s}$), the LPIM approach uses a plunger system or compressed gas principle to produce green parts using pressures varying from 0.1 to 1 MPa [8–10]. Although the binder system drives the mixing, injection, and debinding stages, the final sintered properties are primarily a function of the powder characteristics, such as the particle morphology, the particle size, and the particle size distribution [11,12]. In MIM, fine spherical powders produced mainly by gas atomization or chemical decomposition with a D_{50} , which is generally $< 20 \text{ }\mu\text{m}$, are routinely used to achieve higher packing densities, better flowability, higher solid loadings, and better sinterability, as compared to irregularly shaped powders [13,14]. However, powders containing coarse irregular particles typically produced by water atomization lead to a higher brown strength (i.e., the strength of the part after debinding), as compared to spherical powders, and allow the manufacture of larger complex shape components, while reducing the overall production cost [15,16]. Although the influence of particle size on the moldability, debindability, and sinterability of MIM components has been demonstrated for spherical

powders [17,18], the effect of this key parameter for irregular powders has only been addressed by a few research groups, all using high-viscosity feedstocks adapted for conventional HPIM technology.

Hausnerova et al. [19] studied the effect of particle size distribution (PSD) on the rheological behaviour of two hard metal carbide irregular powder lots. They concluded that feedstocks formulated with a bi-modal PSD exhibit lower viscosities and higher solid loading capacities than those formulated with uni-modal PSD. Hu et al. [20] explored the fabrication of MIM parts using three irregular-shaped water atomized stainless steel powders ($19 < D_{50} < 66 \mu\text{m}$). They confirmed that while the feedstocks prepared with finer powders decrease the moldability, their sintering results in parts with well-developed microstructures and fewer interconnected pores, and therefore, with higher densities, enhanced mechanical properties, and better corrosion resistance, as compared to coarser powders. Mukund et al. [21] investigated the effect of PSD on the feedstock viscosity, sintered properties, and dimensional stability of MIM components obtained from water atomized stainless steel powders with irregular and ligamental morphologies. They demonstrated that an increase in the volume fraction of coarse powders, as well as a decrease in the powder sphericity, decrease the flow performance of feedstocks and the overall sintered part quality. Contreras et al. [22] studied the effect of powder size and shape on the green strength, debinding, and sintering behaviour of different bronze-based feedstocks prepared from a mixture of fine spherical gas atomized and coarse irregular water atomized powders at a ratio of 9:1, respectively. They determined that while the green strength is mostly driven by PSD, and not by particle morphology, an addition of irregular water atomized powders increases the tortuosity of the path through which the binder travels to leave the part, thus decreasing the debinding rate. Notwithstanding this last phenomenon, they

obtained sintered densities and mechanical properties similar to those of parts produced from only fine spherical gas atomized powders. Similarly, Shu and Hwang [23] used different iron-based powder mixtures constituted from coarse irregular (water atomized) and fine spherical (chemically decomposed) powders to investigate the effect of particle size and shape on the feedstock moldability and powder sinterability. They concluded that replacing fine spherical powders with coarse irregular powders (ratio of 6:4) does not significantly change the moulding and debinding behaviour of iron-based feedstocks. On the other hand, adding small quantities of α -phase stabilizers, such as P and Mo, minimizes the grain growth and improves the relative sintered density up to 96%, even for a powder mixture containing up to 60 wt. % of coarse irregular powders.

Equally, in a bid to lower the raw material costs by using irregular powders, some research groups also explored the fabrication of MIM parts using recycled materials. In this context, Rane and Date [24] investigated the rheological behaviour of two feedstocks, one prepared from irregular-shaped fine powders produced by grinding sludge, and another one obtained by blending the first powder with fine graphite flakes. They confirmed that feedstock formulated with only recycled powder manifests low flowability due to high inter-particle friction inherent to the powder shape, while the addition of graphite flakes decreases the viscosity and improves the moldability of such low-cost feedstocks. A few years later, the same research group confirmed that recycled iron oxide powders can potentially be used in the production of low-cost MIM components despite lower achievable solid loadings and sintered densities as compared to virgin powders [25].

Notwithstanding the efforts mentioned above, the fabrication of MIM parts from irregular metallic-based feedstocks has generally been realized using only the HPIM route, while the use of such irregular powders in the LPIM process has never been

demonstrated. To start filling this gap, this work aims to study the effect of powder shape and size on the sintered density of iron-based components produced using the LPIM processing route.

2 Experimental procedure

2.1 Materials and feedstock characterization

Four different feedstocks were prepared using three irregular iron powder lots provided by Rio Tinto Metal Powders (Sorel-Tracy, Canada) and one spherical powder lot. Irregular powders were produced using a three-step workflow comprising water atomization, high-energy milling, and sieving with three mesh sizes: -45 μm , -20 μm , and -10 μm . The spherical powder produced by carbonyl iron powder was referenced as CIP. A scanning electron microscope (Hitachi 3600, secondary electron detector) and a laser diffraction particle size analyser (Beckman Coulter LS13 320) were used to evaluate the powder morphology and quantify the particle size distribution, respectively. The -45 μm , -20 μm , and -10 μm powder lots exhibited irregular and angular morphologies generally expected of powders produced using this powder fabrication workflow (Figure 1a-c), while the CIP showed a mixture of spherical and near-spherical particles typical for this fabrication route (Figure 1d). The frequency and cumulative particle size distributions plotted in Figure 1e-h were used to extract the powder characteristics such as D_{10} , D_{50} , and D_{90} , as reported in Table 1. The density of the metallic powders and binders, as well as the maximum solid loading of each feedstock, were measured using a helium gas pycnometer (AccuPyc II 1340) according to the ASTM B923-16 standard [26].

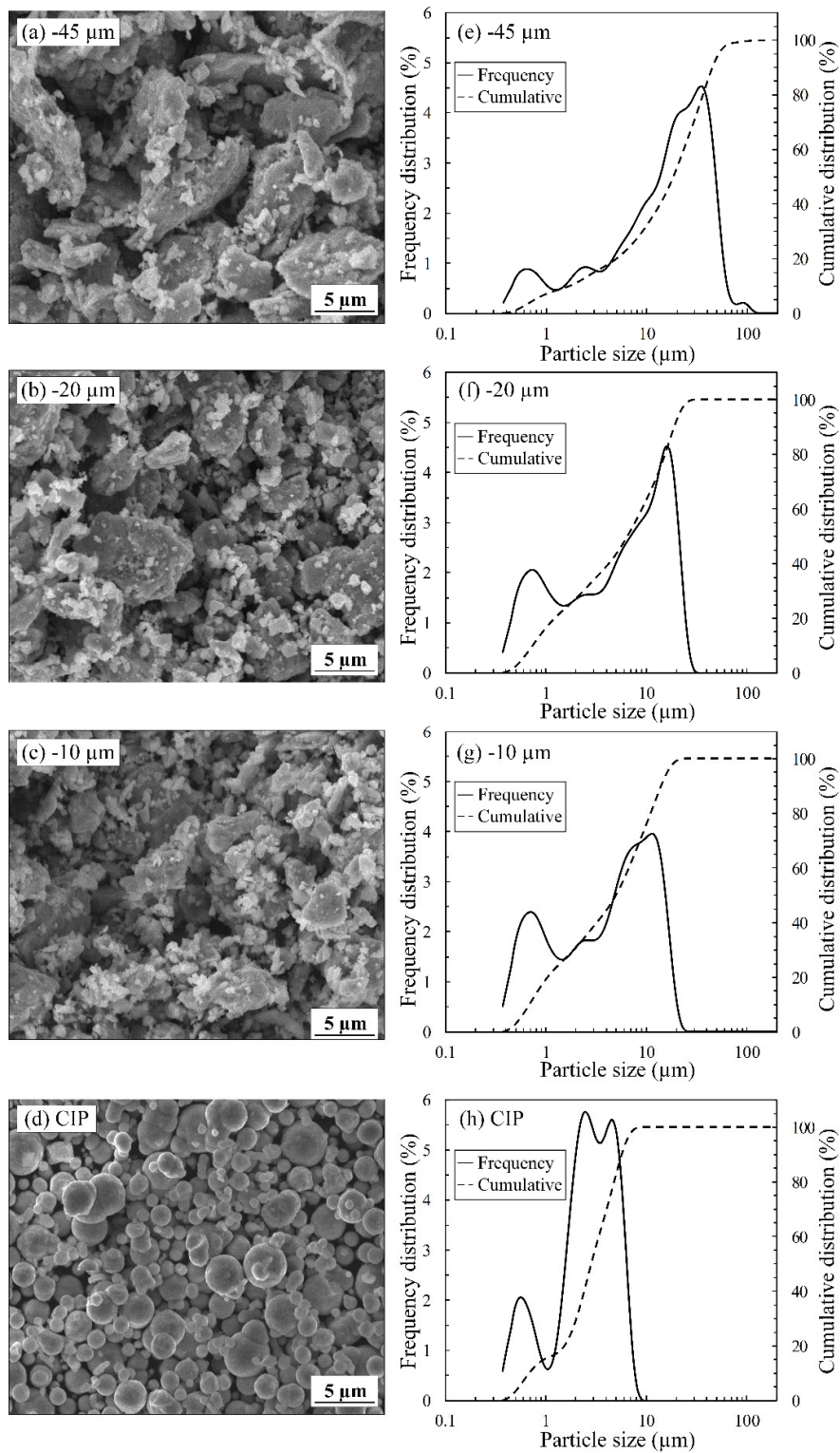


Figure 1. SEM photographs and particle size distributions of four iron-based powder lots: (a, e) -45 μm, (b, f) -20 μm, (c, g) -10 μm, and (d, h) CIP.

Table 1. Characteristics of iron powders.

Powder identification	Shape	D ₁₀ (μm)	D ₅₀ (μm)	D ₉₀ (μm)	Density (g/cm ³)
-45 μm	Irregular	1.9	18.3	44.9	7.4
-20 μm	Irregular	0.7	6.5	19.0	7.1
-10 μm	Irregular	0.7	5.0	14.1	6.9
CIP	Spherical	1.8	4.0	7.7	7.5

The binders were formulated from a blend of paraffin wax (PW) used as the base constituent, and three other additives, including stearic acid (SA) used as a surfactant agent, carnauba wax (CW) used as a shrinking agent, and ethylene-vinyl acetate (EVA) used as a thickening agent, with the characteristics reported in Table 2. These constituents were selected based on their extensive use in the LPIM process [27–29]. Four feedstocks were formulated from the four iron powder lots using similar binder proportions but different solid loadings, each representing the maximum reachable value for each powder lot. Based on recent works on similar water atomized iron-based powders [30], the binder proportion was set at 1 vol. % of SA, 2 vol. % of CW, 2 vol. % of EVA, and the balance of PW, to create adequate surfactant, demoulding, and thickening effects. Dry powders were mixed in a laboratory mixer with the molten binders for 4 hours at 80°C under a vacuum to remove micro-bubbles from the feedstock. Differential scanning calorimetry (DSC) analyses were conducted using a Perkin Elmer Pyris 1 DSC to evaluate the melting points of the binder constituents and feedstocks according to the ASTM D3418-15 standard [31]. To this end, feedstock samples with a mass of around 10 mg were encapsulated in standard aluminium hermetic pans before performing two sequential temperature scans from 30 to 100°C with a heating rate of 10°C/min and dry nitrogen gas flow of 50 ml/min. The

DSC thermograms were recorded from the second heating cycle to minimize the influence of thermal history and maximize the thermal contact between the pan and the specimen.

Table 2. Characteristics of binder constituents.

Constituent of binders	Melting point (°C)	Density (g/cm ³)	Supplier
Paraffin wax (PW)	59.0	0.9	Sigma-Aldrich
Carnauba wax (CW)	84.5	1.0	Sigma-Aldrich
Stearic acid (SA)	75.0	0.9	Sigma-Aldrich
Ethylene-vinyl acetate (EVA)	49.0	1.0	Sigma-Aldrich

The viscosity profiles of the four feedstocks were obtained using an Anton Paar MCR 302 rotational rheometer equipped with a concentric cylinder cell (CC17) and a Peltier temperature-controlled system (C-PTD200). The profiles were obtained at a constant temperature of 80°C for shear deformation rates ranging from 0.5 to 500 s⁻¹. To prevent potential segregation potentially arising during the tests, the feedstock, cylinder, and container were pre-heated to the testing temperature according to the procedure developed in [32]. A thermogravimetric analysis (TGA) was performed using a PerkinElmer STA 8000 to measure the initial and final binder decomposition temperatures. To this end, a specimen of about 1 mm³ was extracted from the injected parts, placed in a standard ceramic pan, and heated from 50 to 550°C using a heating rate of 20°C/min under high purity argon purge gas at a flow rate of 50 ml/min.

2.2 Injection, debinding, and sintering

The LPIM injection press presented in Figure 2a was used to inject each feedstock into the rectangular mould cavity illustrated in Figure 2b. The rectangular green parts (165 ×

12.5 × 3 mm) were produced using a constant volumetric flow rate of 2.2 cm³/s to generate a maximum injection pressure of about 0.5 MPa, with a feedstock temperature maintained at 80°C, and a mould temperature set at 40°C.

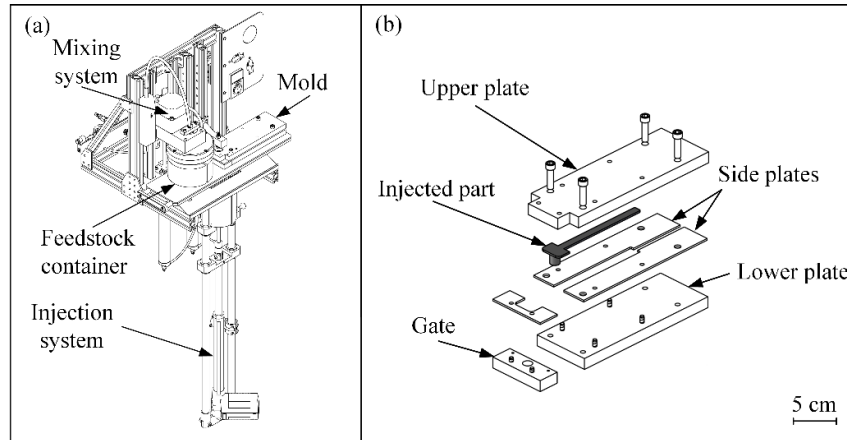


Figure 2. (a) Schematic view of the LPIM injection press, and (b) exploded view of the rectangular mould used during injections.

The green parts were then settled on an alpha-phase 99.9% alumina powder bed (provided by Alfa Aesar, Thermo Fisher Scientific, lot # 42572), compacted in a metallic boat illustrated in Figure 3a, before being buried in loose alumina powder, as shown in Figure 3b. This inert alumina powder was used as a wicking medium during the debinding stage as well as to support the parts before the pre-sintering stage. Metallic boats containing the parts and alumina powder bed were placed into a tight metallic retort in which air was removed using a vacuum pump and replaced with a continuous 15 l/min argon gas flow throughout the debinding pre-sintering cycle. The retort was introduced into the Nabertherm N 41/H debinding furnace to perform the thermal wick-debinding cycle developed in this work. Since the design of a debinding cycle is an important segment of the present work, all details are available in the Results and discussion section.

Following the debinding treatment, the brown parts were removed from the boat, and air-cleaned to remove alumina powder. One part per powder condition was broken

to observe the bonding networks at different locations along the cross-section using a scanning electron microscope (Hitachi 3600, Secondary Electron Detector). The rest of the debound specimens were settled on a dense zirconia plate and sintered in a high-temperature CM Furnace (model 1516 GSH2FL). Using the sintering parameters developed in [33] for iron-based MIM parts, the parts were heated up to 1250°C for 90 min under a pure hydrogen gas flow of about 30 l/min (Figure 3c). The sintered density was measured using the oil impregnation method based on Archimedes' principle according to MPIF standard 42 [34]. Metallographic analyses were performed on cross-sections of the sintered parts, which were vacuum-impregnated by epoxy resin, cured at 60°C, mirror-polished, and observed using an optical microscope in unetched condition. As presented in Figure 3d, the debinding stage produced no significant shrinkage as compared to the injected part, while sintering resulted in a linear shrinkage of about 12%.

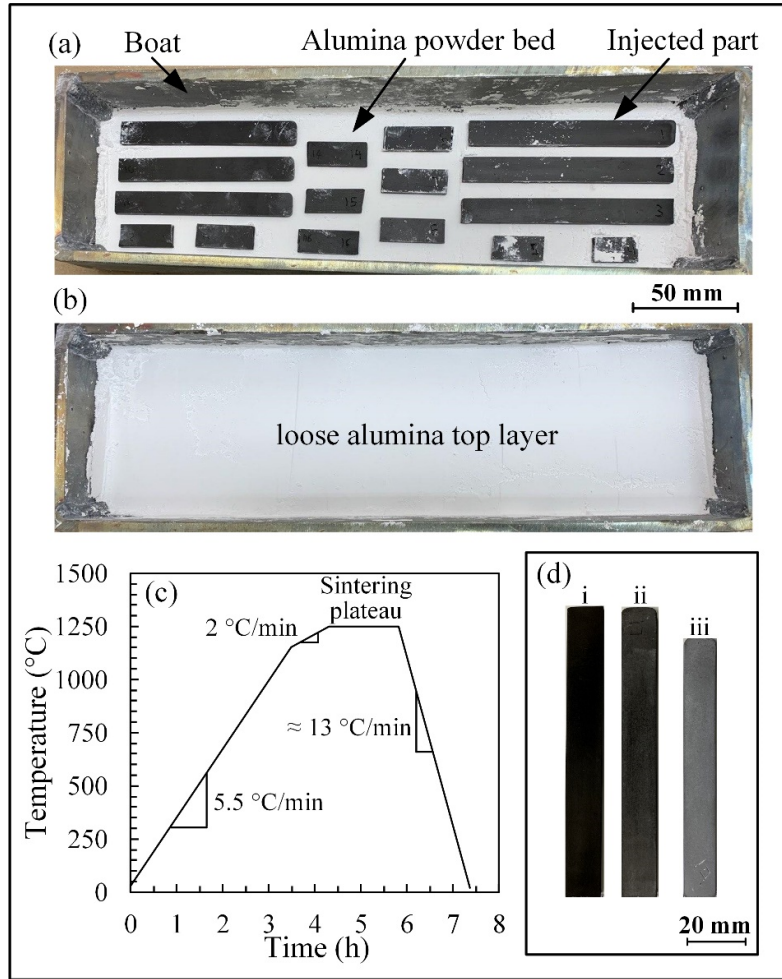


Figure 3. Debinding setup illustrating the (a) injected parts settled on a compacted alumina bed in a boat, (b) parts buried in powder bed, (c) sintering cycle, and (d) overall dimensions of the i-injected, ii-debound, and iii-sintered.

3 Results and discussion

3.1 Evaluation of the maximum solid loading, melting point, and viscosity of feedstocks

It is expected that the higher the volume fraction of powder in the feedstock (also called solid loading), the smaller the shrinkage occurring during sintering. For a given feedstock, this value is set close to or slightly below the critical solid loading providing the necessary lubricity for moulding. Since each powder and binder combination exhibits different powder loading capabilities, the maximum solid loadings for each

feedstock were assessed from the pycnometer density profiles reported in Figure 4. Each feedstock density was measured three times using different samples and producing a standard deviation as low as $\sim 0.003 \text{ g/cm}^3$ (not shown in Figure 4, since the error bars are smaller than the marks). In this work, the critical solid loading was determined according to the criterion where the measured density diverged by 0.025 g/cm^3 (dashed line) from the theoretical density (continuous line). For the same powder shape, the coarser the powder, the higher the critical solid loading, which explains why the measured densities of the $-45 \text{ }\mu\text{m}$, $-20 \text{ }\mu\text{m}$, and $-10 \text{ }\mu\text{m}$ feedstocks diverged from their theoretical equivalents at ~ 59 , 59 , and $57 \text{ vol. \%}$ of powder loading, respectively. Due to its higher sphericity, the feedstock formulated with spherical CIP particles exhibited a critical solid loading of $\sim 63 \text{ vol. \%}$, which is significantly higher than that of the finest $-10 \text{ }\mu\text{m}$ irregular powder. Since preliminary injection results confirmed that the feedstocks of this study were difficult to inject even into a simple shape cavity, the maximum solid loading used in this work was set at $1 \text{ vol. \%}$ below the critical solid loading, as indicated by white arrows in Figure 4 and reported in Table 3. The solid loading values presented herein were determined at room temperature, and their polymer proportion references the feedstock identification. For example, the feedstock 37PW-1SA-2CW-2EVA reported in Table 3 is a mixture containing $58 \text{ vol. \%}$ of powder with $37 \text{ vol. \%}$ of paraffin wax, $1 \text{ vol. \%}$ of stearic acid, $2 \text{ vol. \%}$ of carnauba wax, and $2 \text{ vol. \%}$ of ethylene-vinyl acetate.

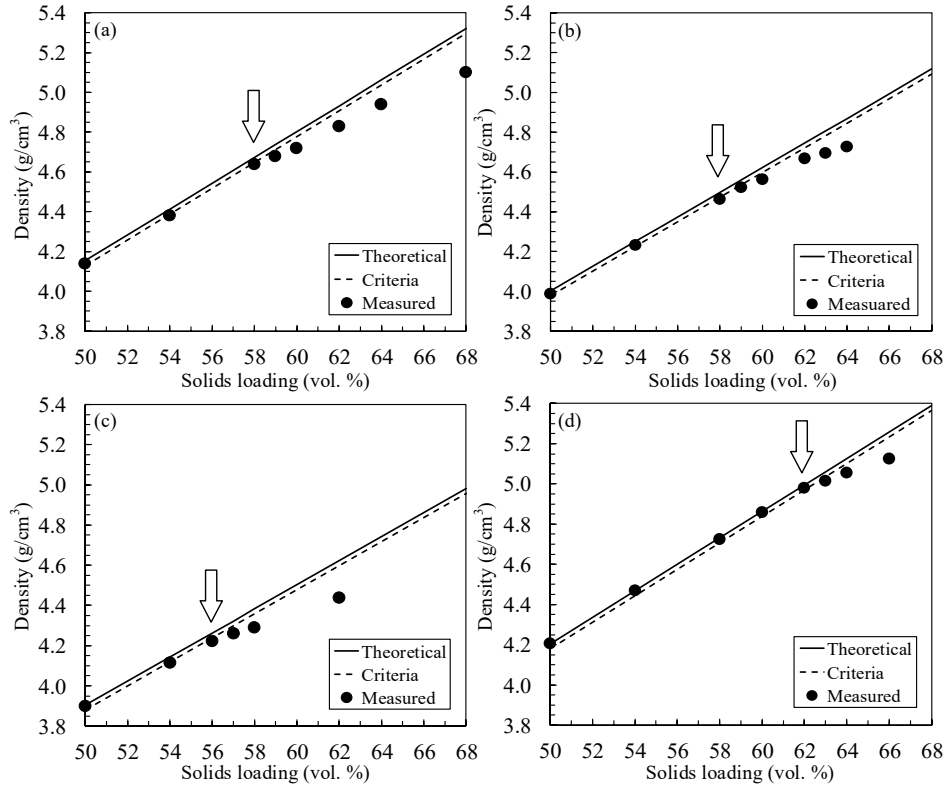


Figure 4. Determination of the critical and maximum solid loadings of the feedstocks prepared with (a) -45 μm , (b) -20 μm , (c) -10 μm , and (d) CIP powders.

The melting points of the feedstocks were determined from the DSC peaks (2nd heating cycle), as indicated by black arrows at 58.5°C in Figure 5a. As expected, the powder characteristics had no impact on the feedstock melting point since this thermal property depends only on the binder formulation, which was similar for all the powder-binder mixtures. Based on this value, the temperature for the rheological tests and injection stage was set at 80°C to guarantee the molten state of the feedstocks, with adequate flowability. Other DSC peaks observed at lower temperatures, indicated by white arrows in Figure 5a, confirm that this multi-binder formulation exhibits several melting points. The DSC graphs also confirm that these binder constituents have a certain degree of solubility wherein the binder blend exhibits a melting point somewhere between the minimum and maximum values obtained for the single constituents (see Table 2).

The viscosity profiles obtained at 80°C for the four feedstocks are shown in Figure 5b. During injection into a simple shape cavity, our previous work [35] demonstrated that the typical shear rate experienced by the LPIM feedstock varies between 10 and 100 s⁻¹. In this range, a viscosity below 20 Pa·s was measured for all feedstocks, i.e., the value proposed by Hidalgo et al. [36] for a typical LPIM injection. Moreover, a shear thinning behaviour was observed for all the feedstocks, with the viscosity decreasing gradually from a low to a high shear strain rate. This well-known phenomenon is attributable to the breaking up of agglomerates, particle ordering, and binder molecular chain alignment with the flow as the speed of the test increases [37]. This rheological behaviour is generally suitable for the LPIM process since having a low viscosity at higher shear rates promotes moldability during the injection stage, while having a high viscosity at low or zero shear rates minimizes segregation during the solidification stage. As expected, the lowest and the highest viscosity values were obtained with the feedstock containing the coarsest (-45 µm) and the finest (CIP) powders, respectively.

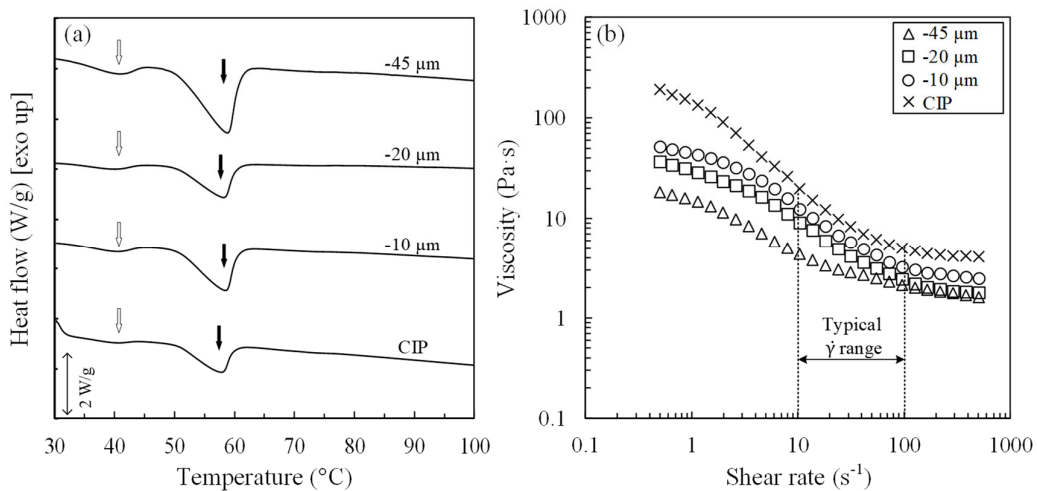


Figure 5. (a) DSC results obtained during heating (2nd cycle), (b) viscosity profile obtained at 80°C for feedstocks prepared with -45 µm, -20 µm, -10 µm, and CIP powders.

Table 3. Characteristics of the feedstocks.

Powder	Maximum solid loading (vol. %)	Feedstock identification
-45 μm	58	37PW-1SA-2CW-2EVA
-20 μm	58	37PW-1SA-2CW-2EVA
-10 μm	56	39PW-1SA-2CW-2EVA
CIP	62	33PW-1SA-2CW-2EVA

3.2 *Design of the debinding cycle and characterization of the debound parts*

The TGA profiles were used to determine the initial and final binder decomposition temperatures presented in Figure 6a. During heating, the weight loss occurring around 250°C (noted as T_{D1} in Figure 6a) corresponds to the beginning of the binder thermal decomposition, which mainly takes place up to 380°C (noted as T_{D2} in Figure 6a), and is completed at 460°C (noted as T_{D3} in Figure 6a). Thermal wick-debinding cycles have three sequential segments: first, to extract the molten binder from the injected part; then, to burn the binder out of the part, and finally, to pre-sinter the powder particles to ensure safe handling of the parts up to the sintering cycle. During the first segment, noted as the debinding plateau in Figure 6b, the temperature generally ranges from T_m to T_{D1} to guarantee a molten state of the binder, while avoiding its thermal decomposition. According to the DSC and TGA tests, the temperature for the debinding plateau was set at 250°C. Furthermore, a low heating rate of about 0.4°C/min, along with a stabilization dwell time of 2 hours, were used to produce a slow and gradual wicking process of the liquid binder (over ~12 hours) and to minimize the temperature gradients that may generate defects, cracks, or blistering inside green parts [38]. During the second segment, noted as the burnout ramp in Figure 6b, the liquid binder absorbed

by the alumina powder bed surrounding the parts was burned out during a T_{D1} to T_{D3} temperature ramp (established from TGA results), using a heating rate as low as $0.8^{\circ}\text{C}/\text{min}$, to minimize the temperature gradient within the debound parts. The third segment, noted as the pre-sintering plateau in Figure 6b, corresponds to a temperature that triggers the solid-state diffusion process and initiates particle bonds, while minimizing powder surface oxidation. According to our previous study [39], the pre-sintering temperature of 650°C is required to ensure sufficient strength for the extraction of all brown parts from the wicking medium. Finally, the debinding furnace was turned off and the specimens were naturally cooled down at approximately $1.6^{\circ}\text{C}/\text{min}$ by maintaining an argon protective atmosphere down to room temperature.

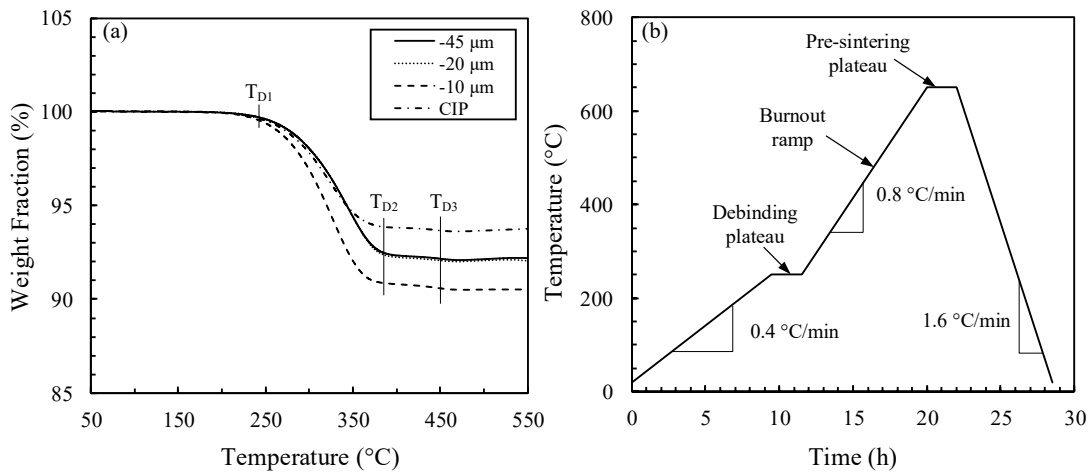


Figure 6. (a) TGA profiles obtained with feedstocks, (b) thermal wick-debinding profile designed based on the DSC and TGA results.

The SEM observations of the debound parts presented in Figure 7 confirm that all the specimens had a large number of open channels (indicated by black arrows in Figure 7a-d), with no visible traces of any remaining binder. However, as it is clearly visible in Figure 7d, these channels were smaller in the CIP parts than in parts produced from other feedstocks. Moreover, no visible particle bonding was observed in the centre of the specimens, irrespective of the powder lot. Nevertheless, as indicated by the white arrows in Figure 7a-c, in specimens prepared from irregular powders, few powder

clusters were observed, thus indicating the possibility of powder bonding (i.e., no clear necking at powder interfaces). Conversely, for the CIP specimens, small particles were well-distributed, and clusters were not visible (Figure 7d). Since brown parts mainly contain a particle network similar to that of loose powders, after this low temperature debinding, their strength is most probably due to an occurrence of solid-state bonds at the surface of the parts and/or the powder particle interlocking.

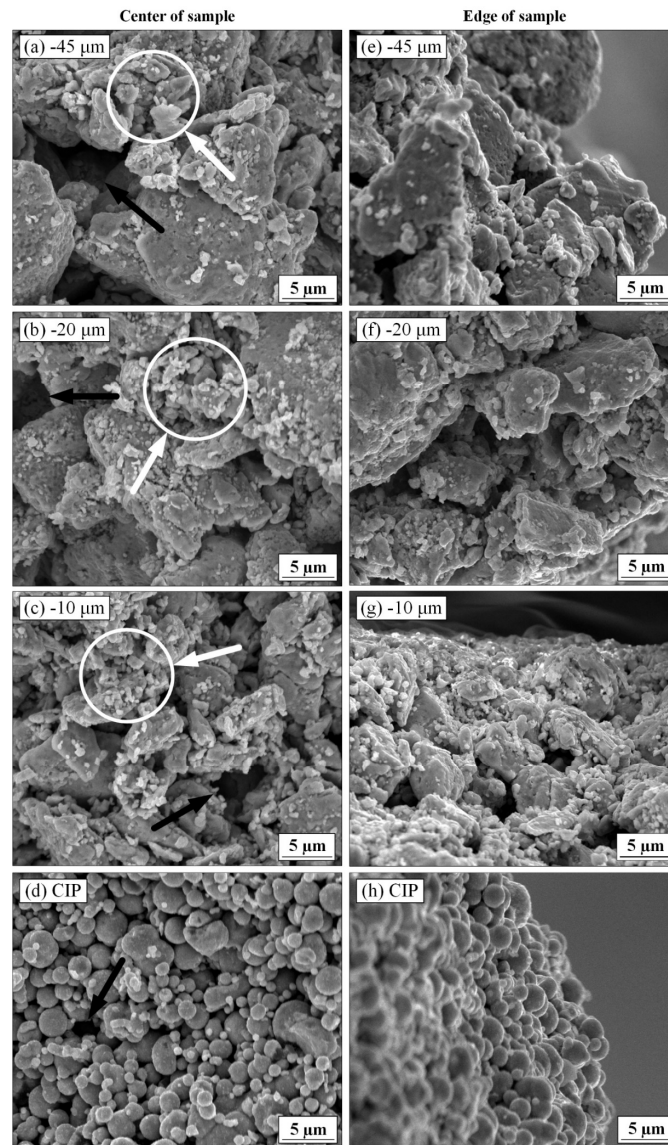


Figure 7. SEM micrographs of the cross-section (a-d) at the center (left column) and (e-h) edge (right column) of the brown specimens produced from -45 μm , -20 μm , -10 μm , and with CIP powders.

3.3 *Sintered properties*

The evolution of the sintered density for the four powder conditions are presented in Figure 8a. For irregular powders, their densities correspond to a density range varying between 6.2 and 6.6 g/cm³, where the coarsest powder (-45 µm) exhibits the highest density value, the midrange powder (-20 µm), the lowest density value, and the finest powder (-10 µm), an intermediate density value. This unexpected trend in density values was confirmed by the metallographic observations. The metallographic observation of the -45 µm powder specimens (Figure 8b) clearly exhibits a higher densification state as compared to the other irregular powders (Figure 8c-d), showing a continuous network of sintered iron (the bright phase in Figure 8b), along with ligamental but almost isolated pores (dark phases in Figure 8b). A decrease in densification capability associated with the lowest density values obtained with the -20 µm powder specimens was also confirmed by a lower amount of densified material combined with a higher number of pores and interconnectivity observed in Figure 8c. The degree of interconnection, as well as the number of pores seen on the metallography for the -10 µm powder specimens (Figure 8d), are between the other two irregular powders, reflecting its density value.

The fact that the most optimal sintered properties for these three irregular powders were obtained with the -45 µm powder specimens could be explained by a more intricate mechanism hindering the sintering, such as an increase in the oxide or soot content, as the size of irregular powder particles decreased. As expected, CIP feedstock with a higher initial packing density and solid loading produced the highest sintered density of 7.5 g/cm³, which correspond to 95% of the iron theoretical density. As shown in Figure 8e, the volume fraction of densified material is significantly higher and the proportion of pores is lower as compared to the irregular powders. However, as

reported by other research groups, higher relative density of 98% can be achieved with CIP powder using higher sintering temperature [40]. This sintering process would need to be further optimized for each of the powder feedstocks used in this study to confirm if higher densification are achievable.

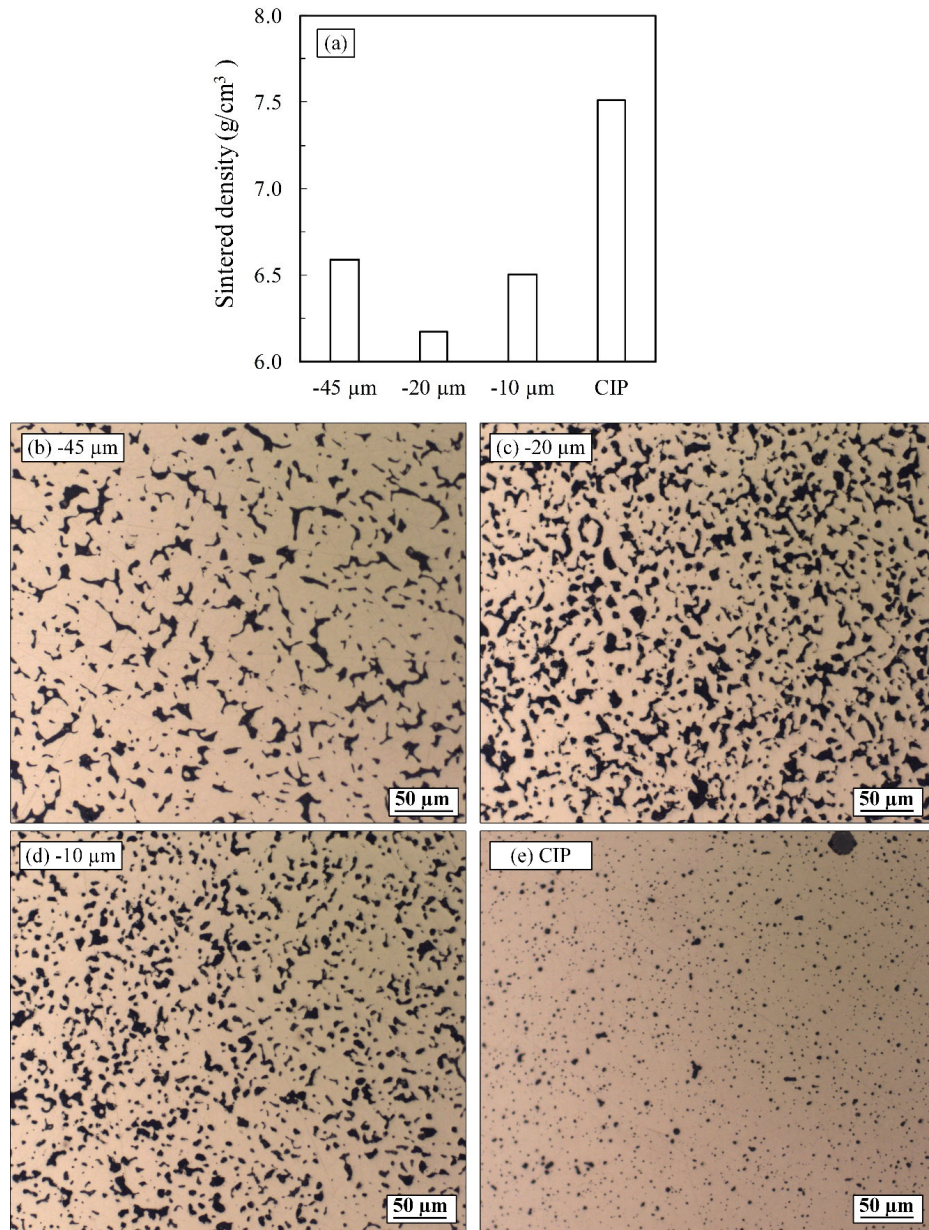


Figure 8. (a) Sintered density and optical micrographs of (b) -45 µm, (c) -20 µm, (d) -10 µm, (e) CIP as-polished specimens after sintering (bright phase: iron, and black phase: pores).

4 Conclusions

Iron parts were produced via the low-pressure powder injection moulding (LPIM) technique using irregular and spherical iron-based feedstocks. Four low-viscosity feedstocks were formulated from three different irregular ($5.0 < D_{50} < 18.3 \mu\text{m}$) powders and one spherical ($D_{50} = 4 \mu\text{m}$) iron-based powder using a common binder system containing paraffin wax, carnauba wax, stearic acid, and ethylene-vinyl acetate. The four feedstocks were injected into a rectangular mould cavity, thermally wick-debound (debinding plateau at 250°C and pre-sintering plateau at 650°C under argon atmosphere for 30 h) and sintered (1250°C under a hydrogen atmosphere for 90 min). The conclusions are summarized as follows:

- The low-viscosity and low melting point ($\eta < 20 \text{ Pa}\cdot\text{s}$; $T_m \approx 59^{\circ}\text{C}$) of feedstocks confirm that the irregular iron powders sieved at -45 , -20 , and $-10 \mu\text{m}$, as well as the spherical iron powder with a D_{50} as low as $4 \mu\text{m}$, can be shaped via the LPIM process.
- The maximum solid loadings obtained with irregular (56-58 vol. %) and spherical (62 vol. %) powders represent the expected values for these two powder morphologies.
- CIP powder exhibits the highest sintered density of $\sim 7.5 \text{ g/cm}^3$. Among the irregular powders, the $-45 \mu\text{m}$ powder produces the highest sintered density at $\sim 6.6 \text{ g/cm}^3$. The metallographic observations confirm these density values.
- To the best of the authors' knowledge, the low-pressure powder injection moulding has been used, for the first time in this study, to produce iron parts from irregular iron powders.

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References

- [1] Atre SV, Weaver TJ, German RM. Injection molding of metals and ceramics. SAE Technical Paper; 1998.
- [2] Heaney DF. Handbook of metal injection molding. Woodhead Publishing; 2018.
- [3] Kryachek VM. Injection moulding. Powder Metall Met Ceram. 2004;43:336–348.
- [4] German RM. Powder injection molding, MPIF. Princeton, New Jersey. 1990.
- [5] Piotter V, Bauer W, Knitter R, et al. Powder injection moulding of metallic and ceramic micro parts. Microsyst Technol. 2011;17:251–263.
- [6] Ruprecht R, Gietzelt T, Müller K, et al. Injection molding of microstructured components from plastics, metals and ceramics. Microsyst Technol. 2002;8:351–358.
- [7] Mangels JA. Low-pressure injection molding. Am Ceram Soc Bull States. 1994;73:37–41.
- [8] Zorzi JE, Perottoni CA, Da Jornada JAH. Hard-skin development during binder removal from Al₂O₃-based green ceramic bodies. J Mater Sci. 2002;37:1801–1807.
- [9] Liberati JF, Araujo Filho OO, Monteiro WA, et al. Low-pressure injection molding processing of AISI T15 high speed steel powders. Mater Sci Forum. 2006;514–516:569–573.
- [10] Gonçalves AC. Metallic powder injection molding using low pressure. J Mater Process Technol. 2001;118:193–198.
- [11] Mamen B, Song J, Barriere T, et al. Experimental and numerical analysis of the particle size effect on the densification behaviour of metal injection moulded tungsten parts during sintering. Powder Technol. 2015;270:230–243.
- [12] Koseski RP, Suri P, Earhardt NB, et al. Microstructural evolution of injection molded gas- and water-atomized 316L stainless steel powder during sintering. Mater Sci Eng A. 2005;390:171–177.

- [13] Hausnerova B, Mukund BN, Sanetnik D. Rheological properties of gas and water atomized 17-4PH stainless steel MIM feedstocks: Effect of powder shape and size. *Powder Technol.* 2017;312:152–158.
- [14] Ali M, Ahmad F. Influence of powder loading on rheology and injection molding of Fe-50Ni feedstocks. *Mater Manuf Process.* 2020;00:1–11.
- [15] Park SJ, Wu Y, Heaney DF, et al. Rheological and thermal debinding behaviors in titanium powder injection molding. *Metall Mater Trans A Phys Metall Mater Sci.* 2009;40:215–222.
- [16] Hashikawa R, Osada T, Kudo K, et al. Control the distortion of the large and complex shaped parts by the metal injection molding process. *J. Jpn. Soc. Powder Powder Metallurgy.* 2016;63:473–478.
- [17] Dimitri C, Mohamed S, Thierry B, et al. Influence of particle-size distribution and temperature on the rheological properties of highly concentrated Inconel feedstock alloy 718. *Powder Technol.* 2017;322:273–289.
- [18] Sotomayor ME, Várez A, Levenfeld B. Influence of powder particle size distribution on rheological properties of 316L powder injection moulding feedstocks. *Powder Technol.* 2010;200:30–36.
- [19] Hausnerová B, Sába P, Kubát J. Capillary flow of hard-metal carbide powder compounds. *Int Polym Process.* 1999;14:254–260.
- [20] Hu Y, Li Y, Lou J, et al. Potential of manufacturing low cost MIM stainless steel. *Int J Met Met Phys.* 2018;3:1–7.
- [21] Mukund BN, Hausnerova B. Variation in particle size fraction to optimize metal injection molding of water atomized 17–4PH stainless steel feedstocks. *Powder Technol.* 2020;368:130–136.
- [22] Contreras JM, Jiménez-Morales A, Torralba JM. Fabrication of bronze components by metal injection moulding using powders with different particle characteristics. *J Mater Process Technol.* 2009;209:5618–5625.
- [23] Shu GJ, Hwang KS. High density powder injection molded compacts prepared from a feedstock containing coarse powders. *Mater Trans.* 2004;45:2999–3004.
- [24] Rane KK, Date PP. Rheological investigation of MIM feedstocks for reducing frictional effects during injection moulding. *Adv Mater Res.* 2014;966-967:196–205.
- [25] Rane K, Date P. Evolution of properties of parts during MIM and sintering of recycled oxide particles. *Powder Metall.* 2019;62:133–145.

- [26] ASTM International. Standard test method for metal powder skeletal density by helium or nitrogen. ASTM Int. West Conshohocken, PA: ASTM International; 2016.
- [27] Rei M, Milke EC, Gomes RM, et al. Low-pressure injection molding processing of a 316-L stainless steel feedstock. *Mater Lett*. 2002;52:360–365.
- [28] Amin SYM, Jamaludin KR, Muhamad N. Rheological properties of SS316L MIM feedstock prepared with different particle sizes and powder loadings. *Inst Eng Malaysia*. 2009;71:59–63.
- [29] Demers V, Turenne S, Scalzo O. Impact of binders on viscosity of low-pressure powder injection molded Inconel 718 superalloy. *J Mater Sci*. 2015;50:2893–2902.
- [30] Tafti AA, Demers V, Vachon G, et al. Effect of binder constituents and solids loading on the rheological behavior of irregular iron-based feedstocks. *J Manuf Sci Eng*. 2020;143:1–36.
- [31] ASTM International. D3418-15 Standard test method for transition temperatures and enthalpies of fusion and crystallization of polymers by differential scanning calorimetry. ASTM Stand. Conshohocken, PA: ASTM International; 2015.
- [32] Fareh F, Demers V, Demarquette NR, et al. Influence of segregation on rheological properties of wax-based feedstocks. *Powder Technol*. 2017;320:273–284.
- [33] You WK, Choi JP, Yoon SM, et al. Low temperature powder injection molding of iron micro-nano powder mixture. *Powder Technol*. 2012;228:199–205.
- [34] MPIF Standard. Determination of Density of Compacted or Sintered Metal Powders, 42. Princeton, NJ. 1986;2–6.
- [35] Ben Trad MA, Demers V, Côté R, et al. Numerical simulation and experimental investigation of mold filling and segregation in low-pressure powder injection molding of metallic feedstock. *Adv Powder Technol*. 2020;31:1349–1358.
- [36] Hidalgo J, Jiménez-Morales A, Torralba JM. Torque rheology of zircon feedstocks for powder injection moulding. *J Eur Ceram Soc*. 2012;32:4063–4072.
- [37] Foong ML, Tam KC. Application of polymer technology to metal injection molding (MIM) processing. *Adv Polym Process Oper*. 1998:213–280.

- [38] Thomas-Vielma P, Cervera A, Levenfeld B, et al. Production of alumina parts by powder injection molding with a binder system based on high density polyethylene. *J Eur Ceram Soc.* 2008;28:763–771.
- [39] Tafti AA, Demers V, Majdi SM, et al. Effect of thermal debinding conditions on the sintered density of low-pressure powder injection molded iron parts. *Met.* 2021;11:1–13.
- [40] Shin DS, Jung ID, Kim HJ, et al. Development of powder injection molding process for sintered soft magnet with the addition of Fe-17 at.% P powder. *Met Powder Rep.* 2018;73:38–45.