Manufacturing of micro-components by powder injection moulding process

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Abstract:

The powder injection moulding (PIM) process satisfies the increasing demand to manufacture smaller parts with complex shapes. The related researches in this area have been carried out at FEMTO-ST Institute since a few years. An injectable feedstock formulation composed of polymers and metallic powders has been tested. Micro injection and bi-material injection tests have been carried out. During the bi-material injection test with a three-plate mould configuration, the interface between the two different feedstocks has been well defined. After thermal debinding stage, the correctly debinded components are sintered with different sintering cycles, the resulted components have been successfully realized.

Keywords: powder injection moulding, bi-material injection, 316L stainless steel, feedstock

1 Introduction

Powder-injection moulding (PIM) is a process based on technologies issued from powder metallurgy for producing near-net-shape components. PIM has the ability to process advanced metallic or ceramic powders into complex-shaped parts [1]. In recent years, to satisfy the increasing demands for microparts in different applications fields, micro powder injection moulding (micro-PIM) already represents one of the key technologies to obtain micro-components or to realize micro-structured surfaces [2]. This is related to the high efficiency and the near-net-shape capability to manufacture complex shaped parts of the PIM process. Four similar stages to the ones in PIM process have been included in micro-PIM process: mixing of very fine metallic powders and thermoplastic binders to get feedstock, injection of powder/binder mixtures in the mould micro-cavity, thermal/catalytic or solvent debinding and finally sintering by solid state diffusion.

2 Feedstock formulation

2.1 Powder

In the last recent years, 316L stainless steel powders are the common one employed in injection molded alloys, either gas atomized or water atomized. It results from the fact that these powders offer interesting capabilities to be sintered to high density and its corrosion resistance [3]. Due to the superior strength of the material produced using pre-alloyed powder compared to the master alloyed prepared material [3], two 316L stainless steel powders have been chosen in our tests to perform the proposed analysis.

The density of both powders is 7.9 g.cm^{-3}. The mean particle sizes is equal to 5 µm and 16 µm, the powder particle size distributions are mentioned in table 1, and the SEM photos of these two powders are given in figure 1 as well.

Table 1. Powder particle size distributions of the two 316 L stainless steel powders used in the present study

Powder	D_{10} [µm]	D_{50} [µm]	D_{90} [µm]
316L stainless steel (5 µm)	1.8	3.4	6.0
316L stainless steel (16 µm)	4.1	10.5	21.9

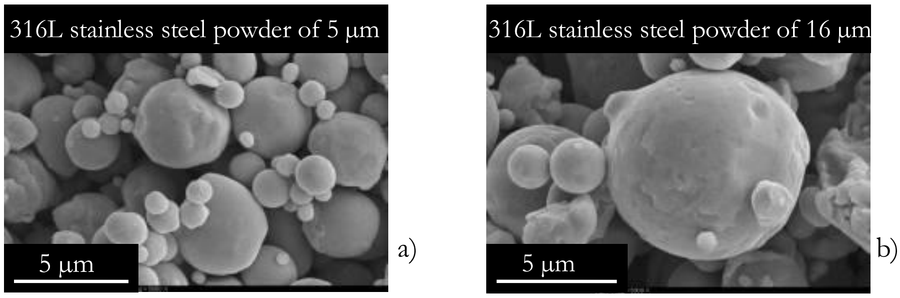

FIG 1. SEM of spherical 316L stainless steel powders used in the proposed investigations

In addition, a copper powder (figure 2) with D_{50} equals 6.34 µm has been used to realize the bi-material-injection experiments, the density is 8.93 $g.cm^{-3}$.

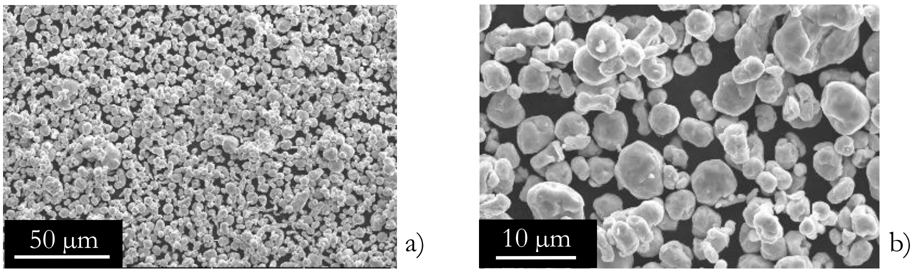

FIG 2. Copper powder (D_{50}=6.34 µm) used in the proposed investigations for bi-material-injection moulding

2.2 Binder

The main role of the binders is to transport the metallic powder particles in the mold cavity during the injection stage, and hold the required shape of the components during the ejection, debinding stage and the beginning of the sintering stage by solid state diffusion.

At present, there are many options for the choice of the binder that is very important in the PIM process. In this investigation, polypropylene (PP), paraffin wax (PW) and stearic acid (SA) has been retained to compose the binder system, due to the fact that the feedstocks prepared according to this feedstock formulation leads to the adapted mixing torque and shear viscosity [4].The descriptions and characterizations of the different binders used to elaborate the feedstocks are related in table 2. Meanwhile, the primary binder PP is used to keep the component shape after injection molding and debinding. The main effect of the secondary binder PW is to decrease the feedstock viscosity and increase the replication ability of the feedstock. The surfactant SA is used to facilitate powder wetting [5].

Table 2. Characteristics and contents of the proposed binder system

Binder	Melting temperature [°C]	Density [$g.cm^{-3}$]	Linear Formula	Comments
PP	140	0.90	$[CH_2CH(CH_3)]_n$	M_w≈304940 M_n≈46832
PW	58~60	0.91	$(CnH_{2n+2})_m$	M_w≈754 M_n≈721
SA	70.1	0.86	$CH_3(CH_2)_{16}COOH$	M_w≈484 M_n≈475

Note: M_w: the weight average molecular weight; M_n: the number average molecular weight.

Table 3. Constituents and related contents used for the three proposed feedstock formulations

Type	316L stainless steel	PP	PW	SA
Volume, %	60	16	22	2.0
Weight, %	92.9	2.8	3.9	0.4
Volume, %	62	15.2	20.9	1.9
Weight, %	93.4	2.7	3.6	0.3
Volume, %	64	14.4	19.8	1.8
Weight, %	93.9	2.5	3.3	0.3

Table 3 summarizes the binder system details according to the proposed formulation; one can notice that the ratio of PP/PW/SA is always 8/11/1. The feedstock (powder volume loading of 60% is taken as an example) related to the proposed formulation has been tested using a twin-screw mixer, according to the following conditions: mixing temperature 160 °C, mixing time 30 min and mixing rotation speed 30 rpm. The mixing torques measured during the tests are illustrated in figure 3 a), on can observe that the final mixing torque corresponding to formulation 2 is about 0.3 N.m. Besides, the viscosity of the feedstock (powder volume loading of 60%) has been measured from capillary rheometer at 200 °C with the length/die diameter (16mm/1mm) ratio always equals 16 and related in figure 3 b). Based on both measurements, the feedstock prepared according to the retained formulation (PP+PW+SA) could be injected.

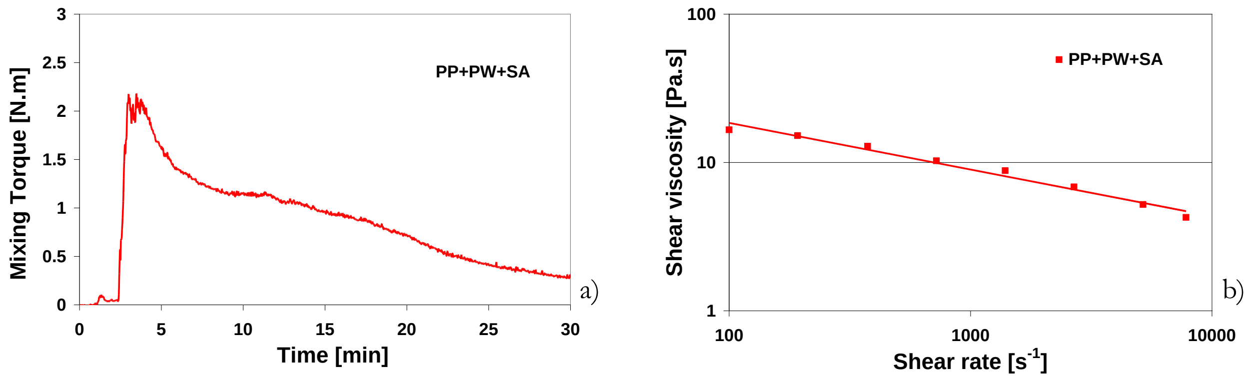

FIG 3. Characterisation of the employed feedstock formulation (powder volume loading of 60%), a) mixing torque vs. mixing time, measured at 160 °C; b) shear viscosity vs. shear rate (100 s^{-1} to 10000 s^{-1})

3 Injection molding tests of selected feedstock

As the injection tests, both mono-material injection with one single feedstock and bi-material injection combining two different feedstocks have been tried by using a Battenfeld® Microsystem micro-injection equipment.

3.1 Mono-injection with micro mould

Four types of specimen have been assembled in a micro mould, the micro die cavity of this two-plate mold is related in figure 4 a), the 316L stainless steel feedstock (powder volume loading of 62%) above prepared is injected and the resulted components are shown in figure 4 b), the thickness of these specimens is 0.5 mm.

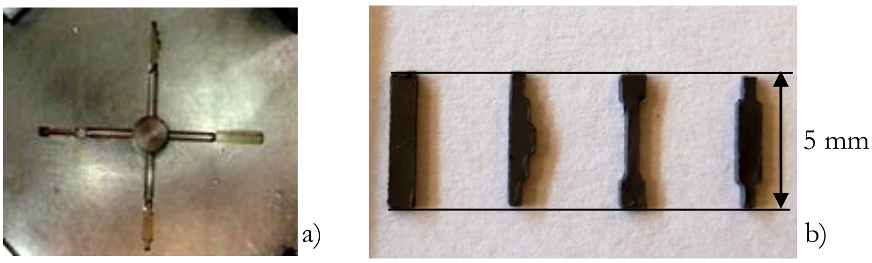

FIG 4. a) Die cavity of the micro mould for mono-injection tests, b) injected micro-components from mono-injection with micro mould (injection temperature: 210 °C, injection volume: 45 mm^3; injection velocity: 10 $m.min^{-1}$; injection pressure: 35 bar)

3.2 Bi-material injection with two-plate mould

Bi-material components could be produced by using co-powder injection molding technique. This process requires a well adapted bi-unit injection molding equipment.

During the first bi-material injection moulding test, the 316L stainless steel feedstock (D_{50}=3.4 µm, according to formulation F3) with different powder loadings of 60% and 64% have been injected into the die cavity of a two-plate mould through two separate sets of sprue and rectangular runner (figure 5 a)) using a Battenfeld Microsystem© 50.

Observing the photo taken under macroscope (figure 5 b)), it is clearly shown that the interface between the two feedstocks is not straight, and its position varies with the injection parameters, this is related to the fact that the die cavities for both feedstocks are not separated, and they are injected simultaneously, so the front flows of both different feedstock flows meet together to form the interface, but this irregular joining of the feedstocks is not able to be repeated uniformly. However, the injected components (figure 5 c)) have been successfully obtained without external defects, and the size and weight of the specimens are uniform.

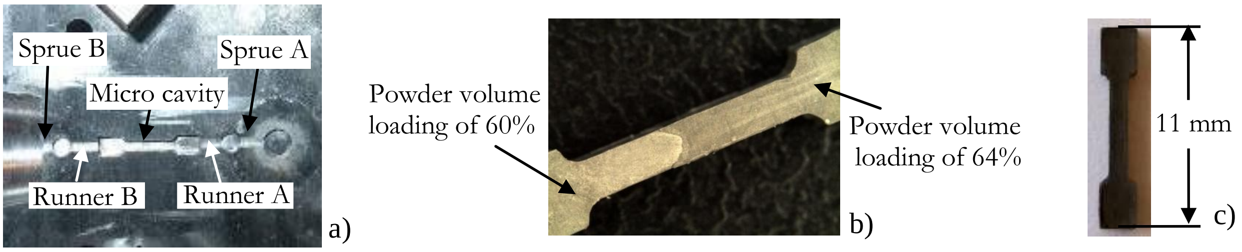

FIG 5. a) Two-plate die cavity mould for bi-material injection test; b) enlargement of the interface region between the two 316L stainless steel (D_{50}=3.4 µm) feedstocks with different powder volume loadings (60% and 64%); c) bi-material injected tensile test specimen (injection temperature: 220 °C, injection volume for both injection units: 30 mm^3, injection pressure: 40 bar)

3.3 Bi-material injection with three-plate mould

In order to solve the joining problem associated to different feedstocks during bi-material injection, another mould (figure 6 a)) specially designed with two separated injection chambers is developed for the bi-material injection test to integrate the 316L stainless steel (16 µm) feedstock (powder volume loading of 60%) and copper feedstock (powder volume loading of 60%) which are prepared according to the proposed formulation.

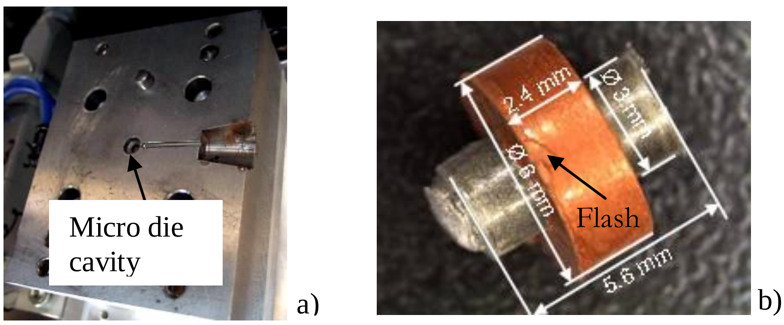

FIG 6. a) Three-plate mould for bi-material injection moulding dedicated to the injection of feedstocks based on 316L stainless steel (16 µm) and copper powder, b) sizes of the injected bi-material component

During the injection tests, the ring has been firstly injected with copper feedstock while a cylinder like pin was inserted into the cylinder die cavity of 316L stainless steel feedstock. And then it was removed to let in the 316L stainless steel feedstock. The interval between both injections is about 0.2 second corresponding to the exit of the internal pin. Thus, the interface has been well controlled. The main injection parameters are the same for both injection units: injection temperature equal 220 °C, injection pressure equal 40 bar, except for the injection volumes equal 45 mm^3 associated to the 316L stainless steel feedstock and 60 mm^3 associated to the Cu feedstock. However, there are still some flashes in edge region (figure 6 b)). The reason of the phenomena is related the loose fits between the moveable and the fixed parts of the mold or over pressurization during mould filling.

4 Debinding tests

During the injection stage, the binder is used to transport out the powder particles to fill out the mould cavities, so it has to be eliminated in the debinding stage. In the present study, thermal debinding process has been set up for the sake of simplicity, safety and environment respect.

A standard thermal debinding cycle has been applied in nitrogen atmosphere. The injected compacts have been firstly heated with a heating rate equal 55 °C/hour from the 20 °C to 130 °C (lower than the beginning temperature of the decomposition of paraffin wax and stearic acid) to vaporize the water vapor absorbed in the injected components. Then the temperature is increased up to 220 °C with a very slow heating rate equal 4.5 °C/hour to eliminate the paraffin wax and stearic acid, without removing the polypropylene which serves as the backbone of the debinded porous components. Finally, the components are cooled during 120 min to reach ambient temperature. During this stage, a porous structure is formed because nearly 75% of the binders are removed. As shown in figure 7, the debinded components corresponding to both mono-injection with micro mould and bi-material injection with two-plate mould after this cycle are free of defects such as uneven binder removal, creating residual stress, etc, and the initial shape of the injected components are well kept.

5 Sintering tests by solid state diffusion

The purpose of sintering stage is to bond the particles together to form a homogenous structure when full densification is achieved [6]. In order to eliminate the pores between powder particles in the debinded components, the porous parts are heated to a temperature slightly lower than the melting temperature of the material used in the process, and then the powder particles bond together under capillary forces.

During the sintering tests for the specimens injected with two 316L stainless steel feedstocks (powder volume loading of 60% and 64%), the temperature has been heated up to 1360 °C to activate fast sintering mechanisms, slightly lower than 1371~1398 °C corresponding to the melting point of 316L stainless steel powders [7]. The related sintering ramps are indicated in table 4, and figure 7 a) show the components after injection, debinding and sintering stage, respectively. The components are correctly sintered with no obvious defects appearing and no cracks near the interface between both different feedstocks, even along the interface zone. In length direction, the sintered bi-material components (feedstock with powder volume loading of 60% and 64%) exhibit over 10.94% shrinkage.

Table 4. Sintering ramps for specimens from bi-material injection

	Ramp 1	Ramp 2	Ramp 3
Heating rate [°C/min]	10	20	-10
Temperature [°C]	600	1360	20
Holding time [min]	30	120	End

Due to the decrease in size (especially the thickness) for the micro-injected components from the micro-injection with fine powder, there is no need for the sintering temperature to be so high (like 1360 °C which has been used in our previous sintering for the bi-material injected specimens). As a temperature about 1250 °C [2] has been proposed in literature, so 1200 °C has been set as the maximal temperature for the micro-mono-injected specimens.

Table 4 gives the details of this sintering cycle and the sintered components issued from this cycle are shown in figure 7 b). As the results, the sintered components obtained are free of obvious defects and 9.6% has been revealed as the mean shrinkage in length direction by the micro-components related to the 316L stainless steel feedstock loaded at 62%.

Table 4. Sintering cycles for specimens from micro-mono-injection

	Ramp 1	Ramp 2	Ramp 3
Heating rate [°C/min]	5	10	-10
Temperature [°C]	600	1200	20
Holding time [min]	30	120	End

In addition, the density after sintering stage of the micro components have been measured by the water displacement method (Archimede's method), a value of 7.51 $g.cm^{-3}$ has been reported as the density, so the relative density has reached to 95.1%.

Besides of the shrinkages and densities of the sintered micro components, Vickers hardness tests have been also used to evaluate the resulting mechanical properties, an average value of HV/160 (before post-treatment like quenching, etc) has been observed.

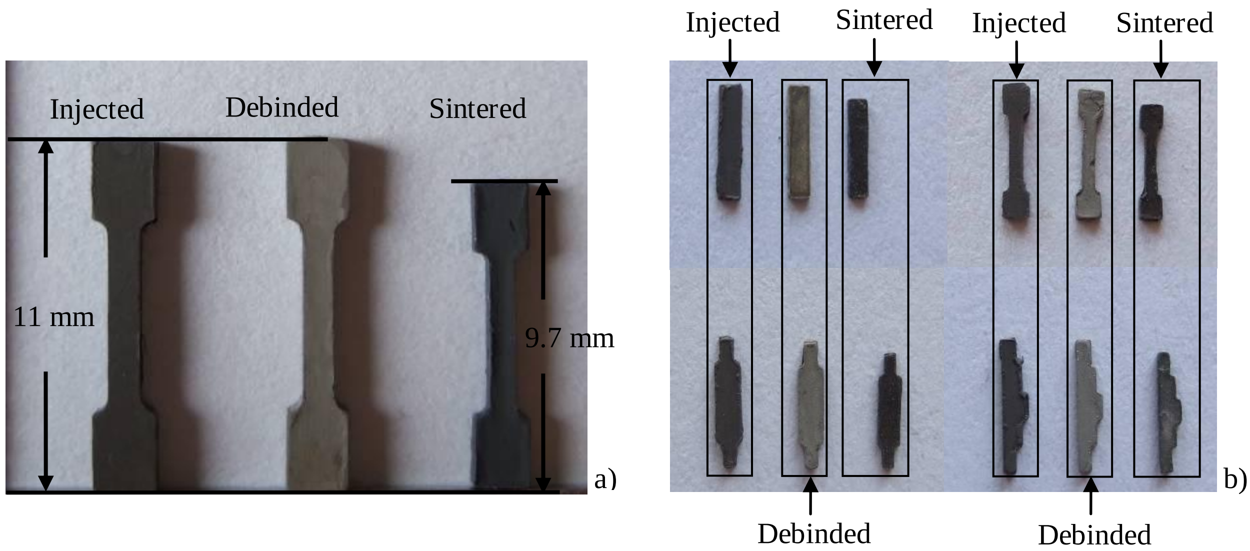

FIG 7. a) Micro-mono-injection specimens, b) bi-material-injection specimens

6 Conclusions

This paper demonstrates a way to manufacture components and microparts by powder injection molding process. By using the proposed feedstock formulation, the feedstocks based on 316L stainless steel powder (5 µm and 16 µm) and copper powder have been obtained, and then injected to realize the micro injection and bi-material injection. During the first bi-material injection test, two 316L stainless steel feedstock with different powder volume loading have been injected using a two-plate mould. In the bi-material injection with the three-plate mould, a 316L stainless steel feedstock and a copper feedstock have been combined, and the interface between the two feedstocks has been well controlled. After thermal debinding stage, the components have been correctly sintered in a furnace and significant shrinkage has been obtained in the sintering stage. The bi-material components injected with two different feedstocks based on 316L stainless steel fine powder (5 µm) have been successfully tested from PIM process.

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