

# Direct observation of mould cavity filling in ceramic injection moulding

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## Abstract

The ceramic injection moulding process is largely used to manufacture 3D net shape components. So far the feedstock behaviours of the mixture of polymer binder and ceramic powder are not well described. In order to better understand the ceramic injection moulding process, a mould with transparent glass windows is manufactured in the laboratory to observe the melt flow during injection stage. The injection process is taken by a high speed CCD camera through the transparent glass windows. In this paper, injection moulding experiments have been performed to observe and compare the filling process, the effects of gate size and injection process parameters on occurrence of jetting have been studied.

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## 1. Introduction

Ceramic injection moulding (CIM) is an efficient process to manufacture small and intricate 3D parts in large quantity under relatively low costs.<sup>1</sup> It includes four basic steps consisting of mixing the ceramics powder and binder, injection moulding,<sup>1</sup> thermal, solvent<sup>2</sup> or catalytic debinding<sup>3,4</sup> and finally liquid phase or solid state sintering.<sup>5</sup> Injection defects such as porosity, welding lines,<sup>6</sup> warping<sup>7</sup> and even cracks<sup>8–10</sup> frequently occur in the injection moulded parts and are commonly related to the binder composition, inhomogeneous mixture, injection moulding parameters and the non-uniform shrinkage during part cooling. This paper presents a detailed analysis of injection process parameters during the ceramics injection moulding, especially focuses on jetting phenomenon.

In ceramic injection moulding, the viscosity behaviour of the powder/binder mixture is greatly different from thermoplastics because the solid volume fraction is as high as about 60% in feedstock.<sup>1</sup> In CIM, the so-called jetting phenomenon occurs, because the mixture hardly exhibits swelling phenomenon near the mould gate, whereas it is typical phenomenon in injection moulding of thermoplastics. The small swelling in CIM is caused

by the slip of CIM feedstock with the mould surface and small elastics recovery out of the mould gate.<sup>10,11</sup> The deformation behaviour of the ceramic mixture is closed to solid rather than fluid. When the jetting phenomenon occurs, the injected feedstock exhibits considerably large filling free surface, this free surface is overlapped during the injection of the CIM feedstock in the mould cavity.<sup>10</sup>

There are two types of jetting described in literature<sup>8,11,12</sup>: conventional liquid state jetting one is and solid state jetting. For conventional jetting, the injected liquid flow coming from mould gate hits its front cavity wall and fills the cavity in sequence of the hit wall to the mould gate. It has an inverse filling sequence of the normal injection moulding in which the cavity near mould gate is firstly fully filled.<sup>11</sup> Conventional jetting results in voids in the final moulded part. Whereas in solid state jetting, one solid finger-like flow enters into cavity from mould gate and curls up itself instead of backward flow in liquid flow jetting. A consequence of solid state jetting is surface faults including weld lines and cracking.<sup>12</sup>

The goal of our investigation is focused on filling phase, the packing and cooling phase have not been studied. In the paper, two different die cavities have been manufactured, they have the same rectangular mould cavity and the same runner length, but different size of gate cross section. The influence of the size of gate cross section on jetting phenomena can be observed by transparent glass windows in the mould.

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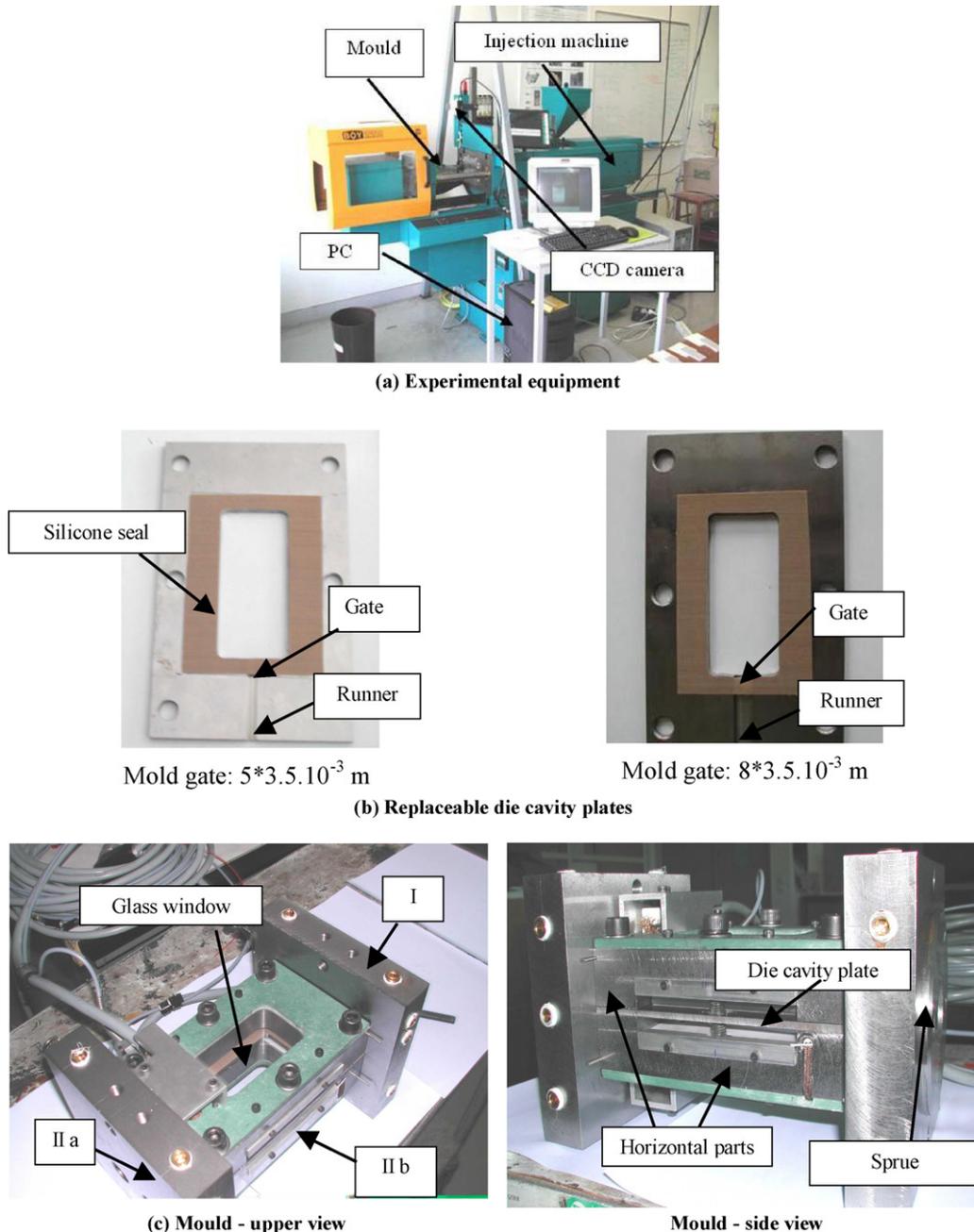


Fig. 1. (a) Testing device, (b) replaceable die cavity plates and (c) transparent mould used in the experiments.

## 2. Experiments

### 2.1. Experimental equipments

Some researches about powder injection moulding have been done in our laboratory.<sup>13–17</sup> CIM experiment in the paper is performed by a Boy 22M/D injection moulding equipment with injection hydraulic pressure control device. The maximum injection pressure is 16 MPa, the screw diameter is  $22 \times 10^{-3}$  m and the injection volume can be varied from 10 to  $30.4 \text{ cm}^3$ . The maximum clamping force is  $22 \times 10^4$  N.

A transparent mould for mould filling real time observations has been designed, manufactured and installed in our labora-

tory to perform the ceramic injection moulding experiments. This transparent mould consists of two principal parts as shown in Fig. 1, in which part I located in injection side and part II locates in fixed side. The part II included one horizontal blocks (IIa) and another (IIb). Between (a) and (b) the replaceable die cavity plate is inserted. Both parts (a, b and die cavity plate) have been connected together by six bolts. The die cavity plate must be manually disassembled after each shot to allow sample ejecting and mould cavity cleaning. The replaceable die cavity plate locates between the two glass plates to enable the observation of the mould cavity filling in real time. All surfaces between metal and glass have been coated with a silicone seal.

The injection mould has a cooling channel for water circulation and six 200 W power heating cartridges, and two thermocouples have been inserted into the mould for temperature control. So it can provide homogeneous mould temperature distribution. The melted CIM feedstock enters in mould cavity from the sprue, runner and gate system in horizontal ways, the mould cavity is fully filled in 0.18 s for various injection process parameters as shown in Table 1. With the use of CCD camera, 25 frame pictures/s were taken through transparent windows of mould. Due to page limit, only a few filling sequential pictures have been selected in this paper.

2.2. Cavity size and injection parameters

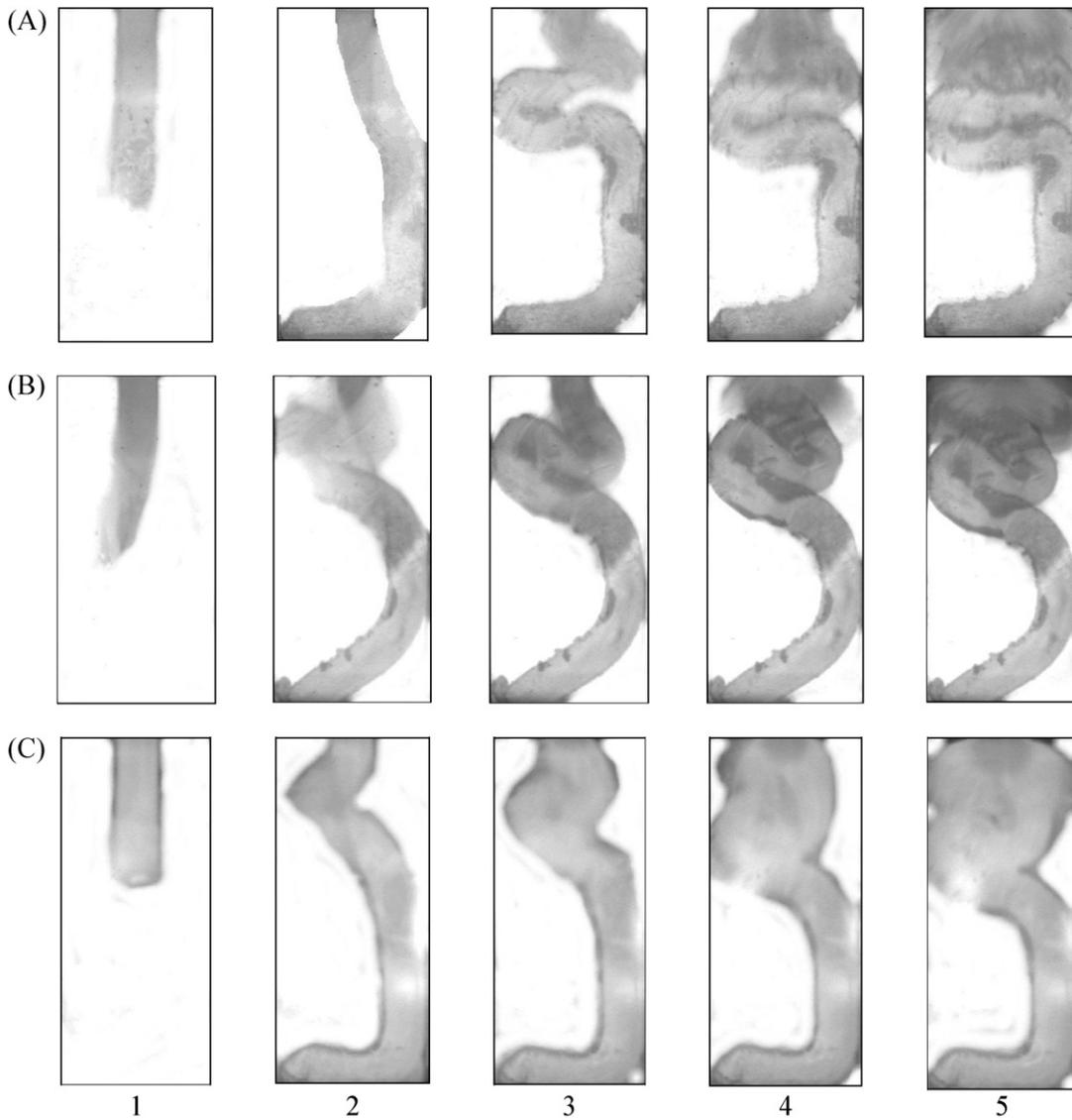
The conic shape sprue with angle of 8° has been manufactured with  $25 \times 10^{-3}$  m length and  $2 \times 10^{-3}$  m initial diameter.

Table 1

Injection parameters used for alumina feedstock in order to compare the effect of melt temperature with mould gate  $8 \times 3.5 \times 10^{-3}$  m

Parameters	Cases			Ref. 18
	A	B	C	
Melt temperature (°C)	200–185	190–185	180–175	165–150
Mould temperature (°C)		60		55 ± 10
Injection pressure (MPa)		14		
Injection velocity (m/s)		$160 \times 10^{-3}$		

The runner length is  $45 \times 10^{-3}$  m. Two gates with same length and different rectangular cross sections have been manufactured. One cross section of gate was  $5 \times 10^{-3} \times 3.5 \times 10^{-3}$  m and another was  $8 \times 10^{-3} \times 3.5 \times 10^{-3}$  m. The size of rectangular die cavity was  $40 \times 10^{-3} \times 90 \times 10^{-3} \times 4 \times 10^{-3}$  m, see Fig. 1. The runner and gate share the same symmetric line of the mould



Mould gate  $8 \times 3.5 \times 10^{-3}$  m.

Fig. 2. Influence of melt temperature in jetting.

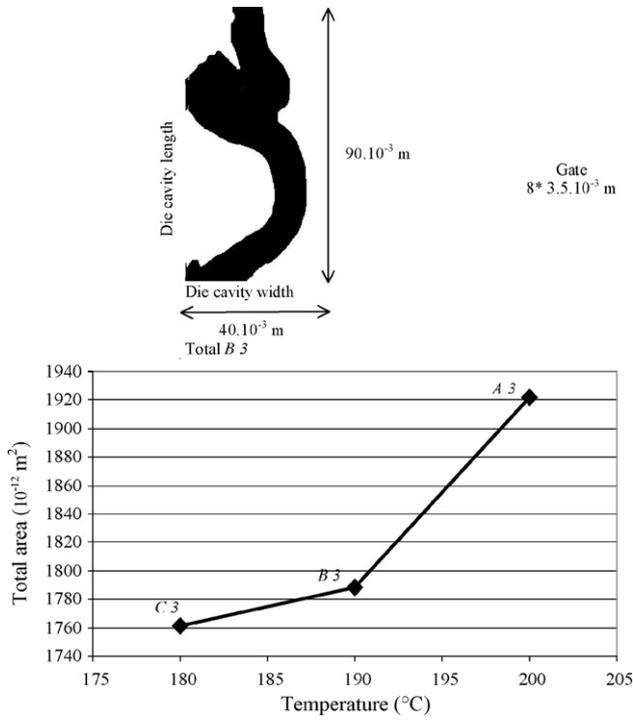


Fig. 3. Dependence of jetting on temperature.

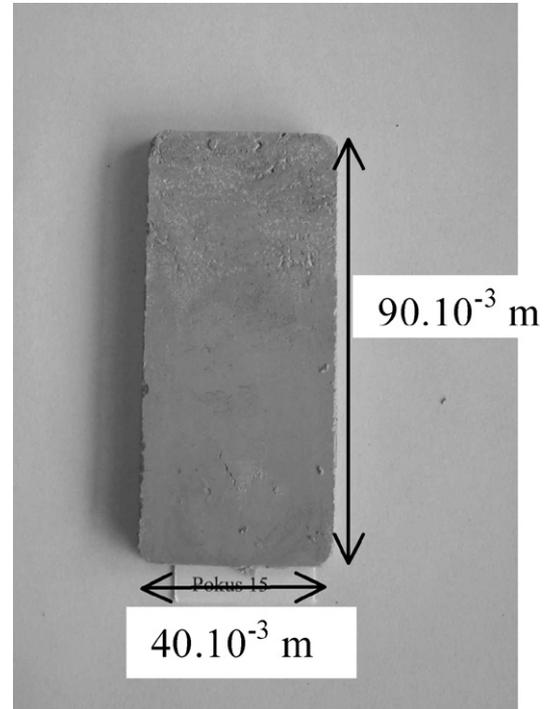


Fig. 4. Moulded component.

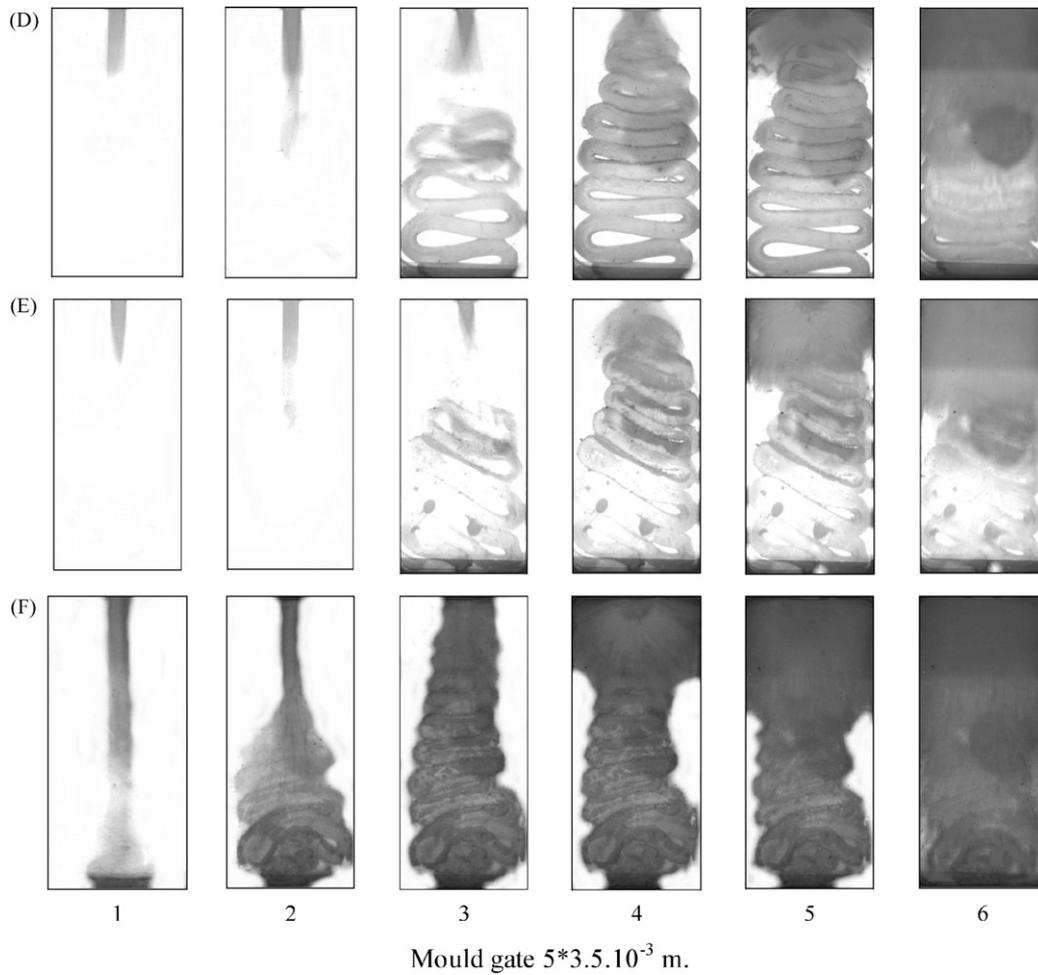


Fig. 5. Projected areas of filling stage.

Table 2

Injection parameters used for alumina feedstock in order to compare the effect of mould temperature and melt temperature with mould gate  $5 \times 3.5 \times 10^{-3}$  m

Parameters	Cases			Ref. 18
	D	E	F	
Mould temperature (°C)	55	60	60	$55 \pm 10$
Melt temperature (°C)	165–150	165–150	185–170	165–150
Injection pressure (MPa)	12			
Injection velocity (m/s)	$120 \times 10^{-3}$			

cavity. This mould cavity is chosen to have the similar shape with literature.<sup>11,12</sup>

The commercially available alumina feedstock ELUTEC® A-99-S provided by Zschimmer and Schwarz was used in the study.<sup>2</sup> It consists of a mixture of alumina powder and binder in the shape of pellets. The fraction of alumina powder in the feedstock was 18.5 wt.%. The binder is the thermoplastic one. The purity of alumina powders was 99.8% and the particle size  $D_{50}$  is equal to  $0.7 \mu\text{m}$ .

The first number of melt temperature correspond to the temperature close the nozzle and the second to the hoper. The temperatures of melted feedstock and mould are chosen similar to those recommended by Zschimmer and Schwarz<sup>18</sup> as shown in Table 1.

### 3. Results and discussion

In order to observe the effect of feedstock temperature on jetting phenomenon, three sets of injection experiments are carried out in the mould with a  $8 \times 3.5 \times 10^{-3}$  m gate. The same feedstock, mould temperature and injection pressure but different feedstock temperatures (melt temperature) have been used for injection experiments, as shown in Table 1. The filling processes pictures at different moments are taken by high speed CCD camera and are shown in Fig. 2. It can be observed in each experiment that a finger like feedstock enter into mould cavity from the gate and touch the opposite mould wall, then curls up. The cured up feedstock accumulates together and form the resistance for newly entering feedstock, so jetting phenomenon can not continue with the increase of the resistance of filled feedstock, as shown in images A4 and B4 in Fig. 2. The lower temperature of feedstock causes the higher viscosity and makes the feedstock properties close to solid one. The experiment C has the lowest temperature in three sets experiments, so it has the highest viscosity, means the lowest fluidity. The low fluidity in the solid-state injection makes

Table 3

Injection parameters used for alumina feedstock in order to compare effect of injection pressure and velocity with mould gate  $5 \times 3.5 \times 10^{-3}$  m

Parameters	Cases			Ref. 18
	G	H	I	
Injection pressure (MPa)	12	10	5	
Injection velocity (m/s)	$120 \times 10^{-3}$	$100 \times 10^{-3}$	$50 \times 10^{-3}$	
Mould temperature (°C)	65			$55 \pm 10$
Melt temperature (°C)	205–185			$165–150 \pm 10$

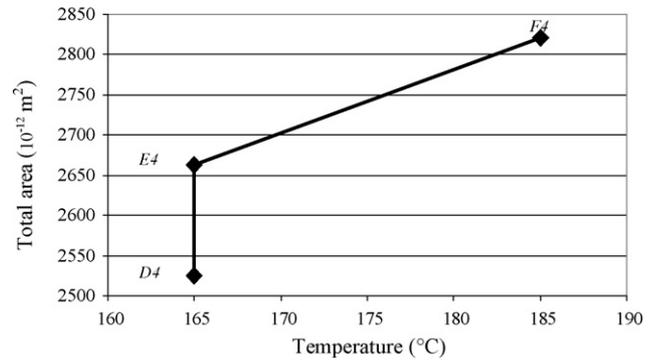
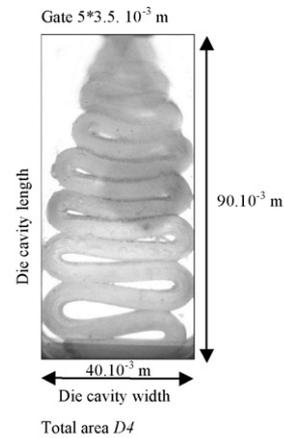


Fig. 6. Trends total area vs. temperature.

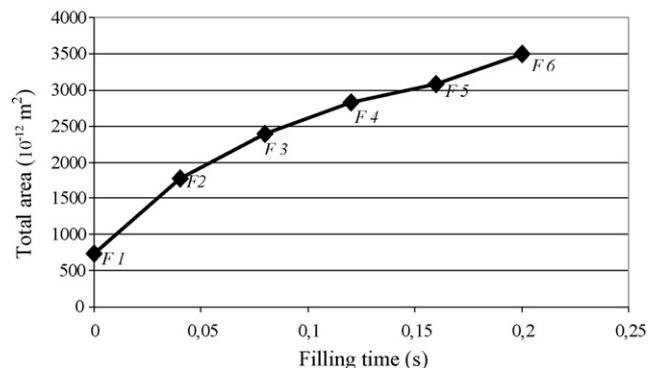
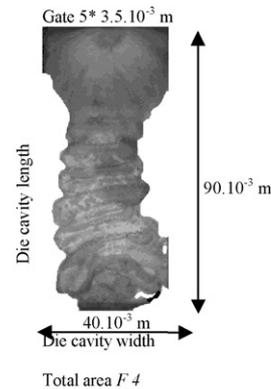


Fig. 7. Development of total area vs. filling time.

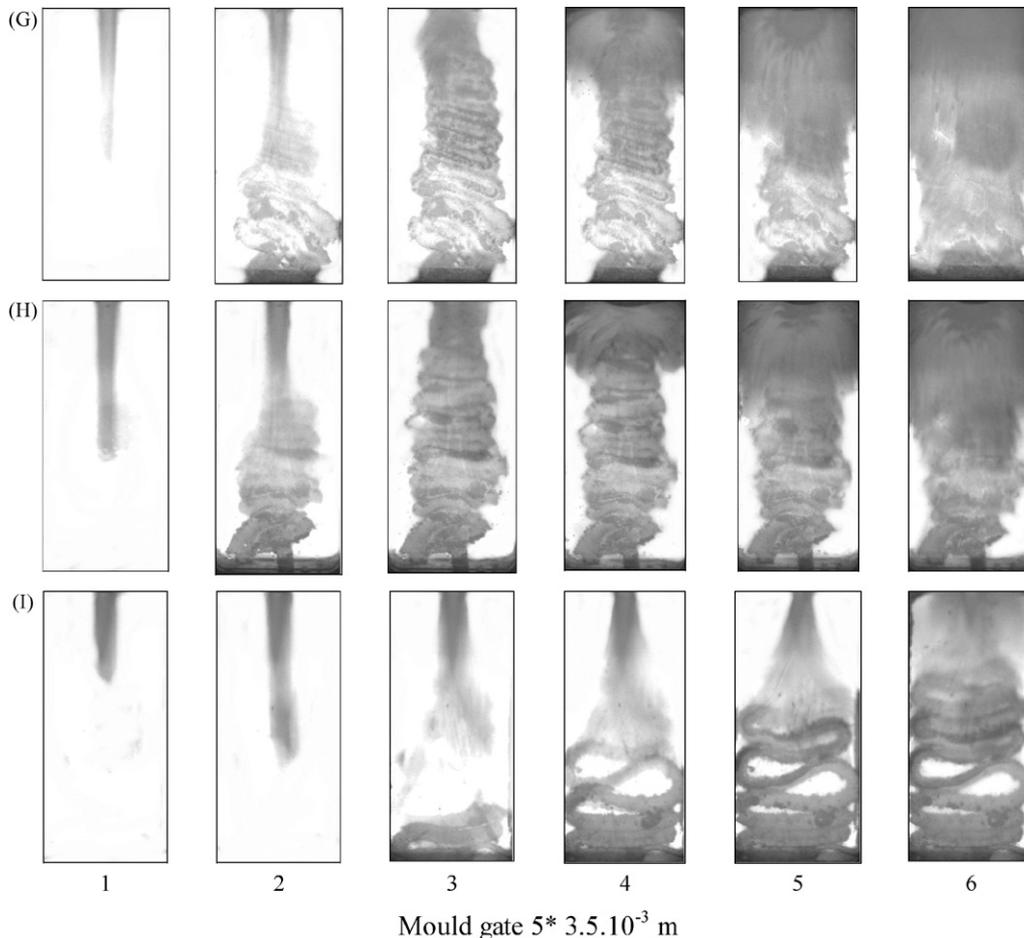


Fig. 8. Projected areas of filling stage.

the jetting difficult to stop, as shown in Fig. 2. In liquid state jetting, higher viscosity or lower fluidity are helpful to avoid jetting, this is the important difference between two kinds of jetting.

With the help of Matlab software to treat the filling process images, the filled feedstock area in the mould can be calculated. As the rectangular mould cavity has the same thickness, so the filled volume can be calculated easily. The comparison of areas filled by feedstock in the mould cavity at the different feedstock temperature is shown in Fig. 3. It shows that 200 °C feedstock can fill much area in the mould cavity than 180 °C one. This is because high feedstock temperature improves its fluidity.

It should be noticed that even after serious jetting as shown in C series pictures of Fig. 2, the part can be obtained without apparent defects, see Fig. 4. However, it is sure that there are some welding lines inside of part, this will cause quality defects in subsequent debinding, especially sintering process. In another hand, jetting has negative effect on the surface quality of part, as shown in Fig. 4.

For the evaluation the mould temperature effect on jetting and the comparison of effect of mould temperature and feedstock temperature on jetting, the three sets of experiments are done in the mould with a  $5 \times 3.5 \times 10^{-3}$  m gate. The injection process parameters are shown in Table 2 and the injection evolution pictures are shown in Fig. 5. It shows that the increase of

mould temperature and feedstock temperature can decrease the area filled by jetting feedstock. As shown in Fig. 6, experiment E has the same feedstock temperature as D one, but it has mould temperature 5 °C higher than D one, it results in  $137 \times 10^{-6}$  m more feedstock filled in the cavity than D one. Experiment F has the same mould temperature as E one, but it has feedstock temperature 20 °C higher than E one, it results in only  $159 \times 10^{-6}$  m more feedstock filled in the cavity than E one. It is obvious that the relative increase of mould temperature make feedstock easily to be filled into the cavity and maybe decrease the possibility of jetting.

Fig. 7 shows that the feedstock is filled into the mould cavity slowly and slowly during injection procedure. It results from the increase of the resistance with more and more feedstock filled in the cavity.

For the evaluation of pressure effect on jetting, the injection process parameters as shown in Table 3 are used for injection moulding, in which the same mould temperature and feedstock temperature, but different pressure are employed for experiments G–I. For experiment G and H using 12 MPa and 10 MPa injection pressure, the jetting domains extend from gate inlet to bottom wall, see picture G2 and H2 in Fig. 8. For experiment I of 5 MPa injection pressure, the domain near gate inlet has not jetting feedstock, see picture I6 in Fig. 8. It means that low injection pressure can avoid the jetting possibility. It is obvious

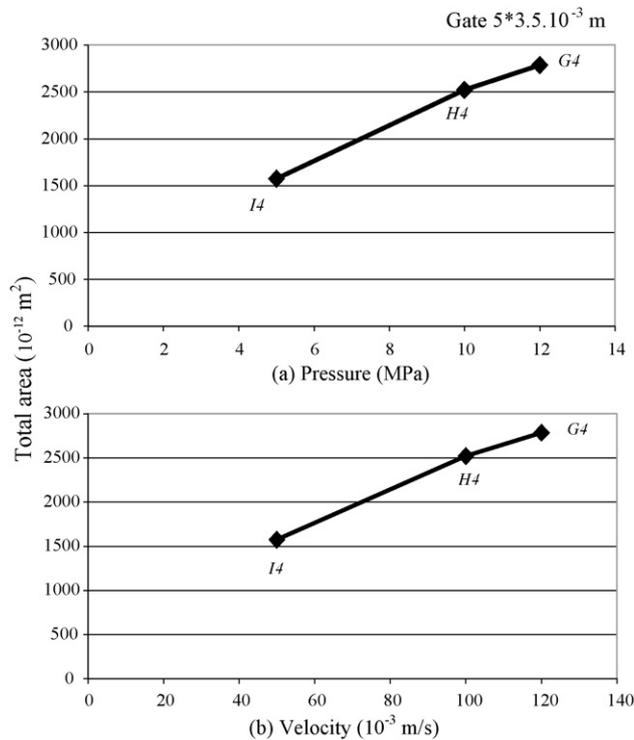


Fig. 9. Trends total area vs. pressure and velocity.

that the injection with high pressure can fill more feedstock in the cavity than low pressure one, see Fig. 9.

#### 4. Conclusion

Various processing parameters during filling phase have been examined on die cavities. The generated phenomenon was continuously recorded and good description of the filling phase was obtained. Firstly, the generation of finger-like steam was observed. Moreover, the jetting occurs in the filling phase around all processing settings. The impression of cases A–C is, that coming down the temperature of melt does not more notably affects this phenomenon on tested geometry of die cavity in our case (gate  $8 \times 3.5 \text{ mm}$ ). Nevertheless, in the experiments employed the wider gate (columns A–D) is fewer jetting than in

narrow gate (column E). The filling stage of ceramic melt with diverse temperature and geometry of the gate were compared.

#### References

- German, R. M. and Bose, A., Injection Molding of Metals and Ceramics, Met. Powder Ind. Federat., Princeton, NJ, 1997, 413.
- Quirnbach, P. and Schwartz, S., Elutec: variable MIM-feedstock systems with aqueous debinding. *Ceram. Forum Int.*, 2004, **11**(81), 23–24.
- Trunec, M. and Cihlar, J., Thermal debinding of injection moulded ceramics. *J. Eur. Ceram. Soc.*, 1997, **17**, 203–209.
- Weinand, D. and Bloemacher, M., MIM feedstock for rapid catalytic debinding. *Met. Powder Rep.*, 1994, **49**(1), 39.
- Heaney, D., Spoilt for choice—commercially available feedstocks for PIM. *Met. Powder Rep.*, 2002, 32–33.
- German, R. M., *Sintering Theory and Practice*. John Wiley & Sons Inc., 1996, ISBN 0-471-05786-X.
- Dubus, M. and Burlet, H., Rheological behaviour of a polymer ceramic blend. *J. Eur. Ceram. Soc.*, 1997, **17**, 191–196.
- Tseng, W. J. and Hsu, Ch. K., Cracking defect and porosity evolution during thermal debinding in ceramic injection molding. *Ceram. Int.*, 1999, **25**, 461–466.
- Tseng, W. J., Warping evolution of injection-molded ceramics. *J. Mater. Process. Technol.*, 2000, **102**, 14–18.
- Krug, S., Evans, J. R. G. and ter Maat, J. H. H., Residual stresses and cracking in large ceramic injection mouldings subjected to different solidification schedules. *J. Eur. Ceram. Soc.*, 2000, **20**, 2535–2541.
- Piccirillo, N. and Lee, D., Jetting in powder injection molding. *Adv. Powder Metall.*, 1991, **2**, 119–126.
- Piccirillo, N. and Lee, D., Jetting phenomenon in powder injection molding. *Int. J. Powder Metall.*, 1992, **28**(1), 13–25.
- Barriere, T., Gelin, J. C. and Liu, B., Experimental and numerical investigations on the properties and quality of parts produced by MIM. *Powder Metall.*, 2001, **44**(3), 228–234.
- Barriere, T., Liu, B., Gelin, J. C. and Experimental, numerical analyses of powder segregation in metal injection moulding. *Met. Powder Rep.*, 2002, **5**(5), 30–33.
- Dvorak, P., Barriere, T. and Gelin, J. C., Jetting in metal injection moulding of 316L stainless steel. *Powder Metall., Maney Publishing*, 2005, **48**(3), pp. 254–260.
- Song, J., Gelin, J. C., Barriere, T. and Liu, B., Experiments and numerical modelling of solid state sintering for 316L stainless steel components. *J. Mater. Process. Technol.*, 2006, **177**(1–3), 352–355.
- Gelin, J. C., Barriere, T. and Song, J., Processing defects and resulting mechanical properties after metal injection molding. *J. Eng. Materials Technology, Trans. ASME, in print*, 2008.
- Zschimmer and Schwartz (Ed.), Technical Leaflet of Siliplast<sup>®</sup>, HO, 2001, pp. 1–12.