## Manufacturing and Investigation of Precision Powder Injection Moulded Parts

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#### Abstract

The improvement of replication accuracy is still an important topic for progress in PIM as it often limits the applicability of the process. Simulation of the filling and cooling steps might help to enhance performance. Another approach is based on a modified process conduct with implemented compression steps which, as further improvement, enables the reduction of membrane thicknesses compared to unmodified powder injection moulding. As an example for precision micro PIM parts ceramic nozzles to be used in experiments with X-ray free-electron lasers are explained. Challenging features are the outlet diameter below 100µm and the internal guiding structures. Such nozzles are required to generate a strongly focused liquid jet of the analysis sample perpendicular to the beam of intense femtosecond X-ray pulses.

#### 1. Objectives

As well-known powder injection moulding provides highly efficient possibilities for manufacturing metal and ceramic parts. The technique has been used since the 1980's completing the conventional methods such as die casting or machining by a technique for fabrication of near-net-shape components. Additional benefits are due to the broad range of possible materials e.g., alloyed steels, hard metals, and high-performance ceramics.

On the other hand it cannot be overseen that there are still particular drawbacks which in certain cases limit the applicability of PIM [1, 2]. One of them concerns the dimensional accuracy of the final sintered parts which often does not fit the strong demands of high value-add products thus costly reworking procedures have to be added [3, 4]. The main reason for this handicap is caused by the hardly controllable sintering procedure, i.e. the parts undergo densification and shrinkage nearly without any dimensional constraints. Therefore, powder-binder segregation, internal stresses and other inhomogenities might cause significant dimensional deviations or even distortions.

To avoid such detrimental effects the product developers have to follow certain lay-out rules while designing the part geometry. One of these rules says that changes of the wall thickness should be kept to a minimum [5, 6]. However, many applications demand designs which do not allow for adherence to this rule. To overcome this complex of problems new approaches have to be started. As one example described in the following chapters, a modified tool technology had been developed which allows for the implementation of additional compressions steps to obtain better dimensional constancies of the final parts.

### 2. Experimental Set-up

To explore possibilities for increased dimensional accuracies of PIM parts even in case of significantly varying wall thicknesses a new experimental set-up had been created: It consists of a relatively broad cylindrical ring covered by a thin disk (membrane section) on the top.

Outer diameter of the cylinders rings in green state was 4.8mm, whereas the membrane thickness could be varied in a range of 600µm to about 100µm depending on the upper/lower position of the movable pistons. These pistons did not only enable the the variation of the volume of the cavity by their up and down movement. Furthermore, after the injection step, the feedstock can be compacted further.

The technical challenge was to produce the membranes on the top of the cylindrical ring as thin as possible and to investigate the thickness reproducibility and the influencing parameters.

### 3. Simulation of the Mould Filling Process

During the phase of component or tool design, particular importance is attached to determine the best design of the gate system and to identify possible critical flow conditions.

The calculations were carried out by means of the software program Autodesk Moldflow<sup>®</sup> using the 17-4PH feedstock material dataset from previous studies.

Whereas the volume of the pressure sensor amounted to  $0.065 \text{cm}^3$ , the volume of the moulded part as a whole was found to be approximately  $1.6 \text{cm}^3$ . In addition, a linear allowance factor of 1.167must be taken into account. In line with previous experience, a melting temperature of  $165^{\circ}$ C and a tool temperature of  $60^{\circ}$ C were assumed for the model calculations. With the aid of an initial simulation the required injection pressure and shear rates for an injection time of 0.55 were estimated. The results showed that it was necessary to increase the injection time up to  $\geq 1$ s. Therefore an injection time of 1.1s was chosen and the changeover from speed to pressure control was performed at 99.5 percent of the mould filling.

The simulation software enables calculation of the temperature distribution during certain stages of the injection moulding process. This allows to identify hot spots caused by excessive friction heat as well as temperature profiles at the time of sealing. Figure 1 shows the temperature distributions for both gating variants after completion of the mould filling process. The resulting heat images are relatively homogeneous. Whereas the four-runners variant causes higher residual temperatures (friction heat) in the voluminous cylinder, the temperature profiles approximate each other again as cooling increases.

The main conclusions to be drawn from the model calculations were the final choice for the fourrunner version and the determination of appropriate gate diameter to avoid extensive shear rates. A more detailed description of the simulation evaluations can be found in [4, 7].

### 4. Experiments and Results

As test material a typical 17-4PH Micro Powder Injection Moulding feedstock had been used. Solid content was 63Vol% of the stainless steel powder with a mean particle diameter of ca. 4.5µm. As binder the so-called GoMikro-system developed at KIT had been applied. It contains 50% paraffine wax, 45% polyethylene and 5% stearic acids plus certain additives.

Trials were performed on an injection moulding machine type Arburg Allrounder 420C-600 with a cylinder diameter of 15 mm.

Complete filling of the membrane by an unmodified PIM process was clearly limited. Several trials including parameter variation showed that thicknesses down to approximately 400µm were feasible, however filling became incomplete in case of smaller gaps,

So operation of the pistons was indispensable and the modified process conduct has to be executed in such manner:

- backward movement of the pistons to open a relatively wide membrane cavity
- inject the feedstock into the wide cavity
- forward movement of the pistons until the final membrane thickness is reached

Debindering and sintering was performed using typical MicroPIM parameter sets, i.e. no special adaption on the particular sample design was necessary. Achieved porosities were in the range of 1.6 - 2.1% with pore sizes of  $1.7 - 2.1\mu$ m.

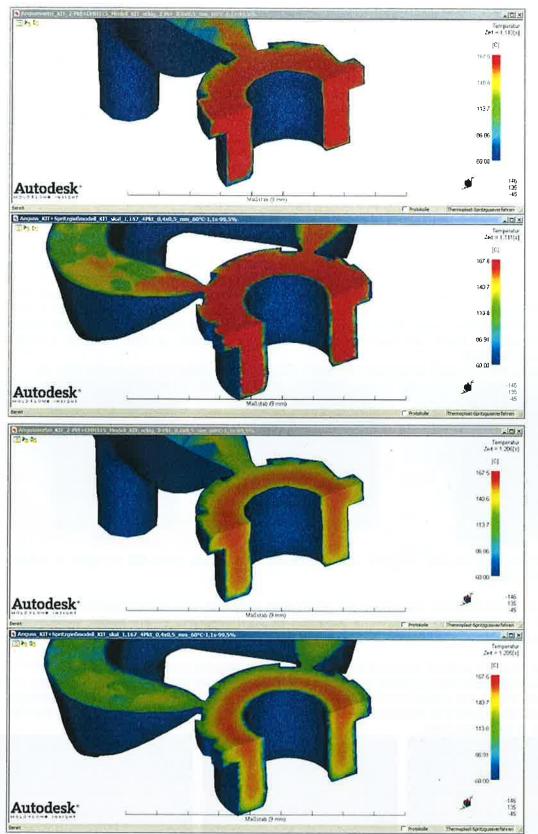


Fig. 1: Calculation of the temperature profile during the back pressure phase as a function of the design of the runner.

First two figures from above: At  $t_1 = 1.112$  s. Last two figures from above: At  $t_2 = 1.205$  s. The feedstock temperature decreases rapidly i.e.,  $\Delta T$  amounts to approximately 10°C of  $t_1 \rightarrow t_2$ . The four-runners version first exhibits slightly higher temperatures which can be assumed to be due to higher friction heating.

One important goal was to investigate the influence of the process parameters on membrane quality. Therefore, a DoE approach covering the most relevant parameters, namely

- the embossing strength (equates to compressing force)
- the gap width, i.e. the distance between dye and ejector piston before embossing, and
- the delay time, i.e. the duration from end of injection until beginning of compression
- had been carried out.

It turned out that best and most reproducible membrane qualities were achieved if highest compression forces (embossing strengths) were used and if the cavity had been opened as wide as possible before injection (gap width). These results verified that the considerations which had led to the injection+embossing process conduct were in principle right.

The embossing delay time, however, showed no significant influence.

Again, a more detailed description of the influence of moulding and compression parameters on membrane accuracy can be found in [4, 7].

The second question to be answered by these trials was the minimum membrane thickness that could be reached in a reliable manner. In contrast to the injection moulding process without compression step the smallest membrane thicknesses achieved were now about  $\leq 200 \mu m$  after sintering. The related variances were approximately ±0.4%. Further trials to achieve even thinner membranes showed that values down to 150 µm are even feasible. If going down to 100 µm (means ca. 90 µm in sintered state) void free membranes could be produced as well, however, a certain waviness was detected.

Besides, green parts of each series were cut open and examined under the microscope (Fig. 2).

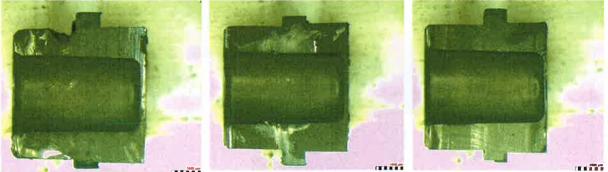


Fig. 2: Images of green-part sections with membrane thicknesses (from left to right) of 200µm, 150µm, and 100µm.

As revealed by the analyses, warping of the green parts increases with falling membrane thickness. This effect may be due to a short-time adhesion of the membrane to the piston surface (it is conceivable that thinner membranes adhere longer than thicker ones and have a lower resistance to distortion).

In the same way, the sintered demonstration objects were examined under the light microscope (Fig. 3). Whereas the green-part specimens with thicknesses of 200µm and 150µm did not reveal any noticeable problems, the 100µm membrane shows some kind of darkening but no conspicuous flaws. As expected, the membrane thicknesses of the sintered parts are about 10 to 13% lower than the thicknesses of the green parts.

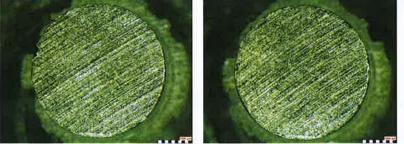




Fig. 3: Light-microscopy images of the outer surfaces after sintering.

# 5. Micro Precision Ceramic Nozzles

Of course, there are various applications requiring micro-sized components which have to meet quite tight tolerances (usually both demands occur in parallel).

As a good example ceramic micro nozzles shall be presented here:

The Center for Free-Electron Laser Science at DESY (Hamburg) and the Institute for Applied Materials at KIT have agreed on a collaborative project to develop ceramic nozzles to be used for X-ray free-electron lasers (such as the LCLS in California or the European XFEL in Hamburg which is currently under construction).

Such specially designed nozzles are required to generate a strongly focused liquid jet of the analysis sample perpendicular to the beam of intense femtosecond pulses of X-rays at the LCLS (Fig. 4). For this purpose KIT has modified its MicroCIM equipment to produce such nozzles with a sophisticated design agreed by both partners (Fig. 5).

First samples have been produced and fabrication experiments and part investigations including spray tests are currently running (Fig. 6).

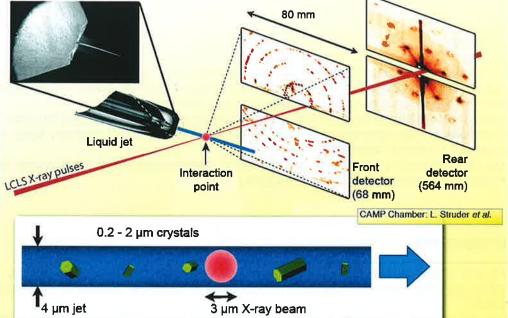


Fig. 4: Operation scheme drawing of the X-ray free-electron laser as it is under construction at DESY.

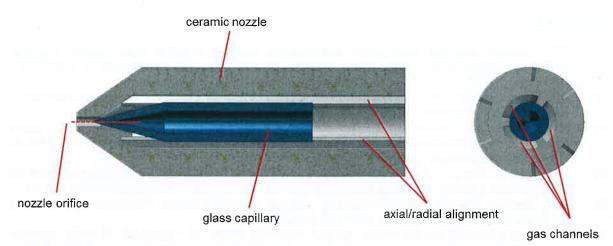


Fig. 5: Schematic drawing of the micro precision ceramic nozzle including the internal alignment features for the precise positioning of the glass capillary in the ceramic body.



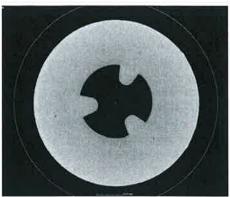


Fig. 6: Pictures of first samples, top view showing the nozzle orifice with a diameter of 100µm (left) and CT image showing the alignment features.

### 6. Outlook

It can be truly stated that dimensional accuracy will represent an important topic for PIM optimization even in future. Main challenges are, for example, the strict limitation of powder-binder segregation, flow control and pressure conduct during the injection and after-pressure phases.

The presented trials showed that additional compression steps offer the possibility to obtain flat and very thin sections of high dimensional accuracy even in combination with relatively bulky volumes, i.e. thorough wall thickness variations become feasible.

Further on, other influencing factors like powder loading, powder composition (multi-modal mixtures), densities and microstructures have to be investigated, too.

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