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Recent developments in micro ceramic injection molding

Dedicated to Professor Dr. Karl-Heinz Zum Gahr on the occasion of his 65th birthday

Effective material application and miniaturization, both indispensable to modern product development and production, demand enhanced manufacturing processes suitable for both micro devices and economic series production. For micro parts made of polymeric materials, micro injection molding represents such a method and has already reached an industrially viable status.

For manufacturing of ceramic products micro powder injection molding is a promising option because it combines the possibility of large-scale series production with a wide range of materials, thus possessing a considerable economic potential.

An enhanced variant, micro two-component injection molding enables, for example, the fabrication of micro components consisting of two ceramic materials with different physical properties and, furthermore, significantly minimizes mounting expenditure.

Keywords: Micro systems technologies; Micro injection molding; Powder injection molding; CIM; Multi-component injection molding

1. Introduction

The market for micro systems products has shown exceptional growth in the past and is predicted to continue to grow in the future. Typical fields of application comprise telecommunication, biotechnology, medical engineering, chemical analysis, microelectronics, and automotive engineering.

Nevertheless, micro manufacturing at present lacks processes suitable for high-strength materials as well as for large-scale production. Therefore, adaptation of the well-established powder injection molding technology to the micro scale is quite a promising option. Development started some years ago and up to now a remarkable technological level, as illustrated in the following sections, has been reached. The present state of the art of micro powder injection molding (MicroPIM) with a clear focus on ceramic materials will be described and some promising special variants will be presented.

2. Micro injection molding

Mainly due to its high economic potential for medium and large series fabrication, injection molding technology is of considerable interest to micro systems engineering. For the adaptation of the process to micro manufacturing, however, additional process features had to be implemented. In this context, tool evacuation and the so-called Variotherm temperature control have to be mentioned. Evacuation of the core section of the tool is necessary as the cavities in a typical micro molding insert represent "bag-type holes" which are not permeable to gases at the bottom. If hot plastic melt was pressed into such a cavity without prior evacuation, the compressed and heated air would cause the organic material to be burned (Diesel effect). Variotherm temperature control means that the molding tool is heated to temperatures near the melting point of the polymer mass prior to its injection into the tool. As a result, flowability of the plastic melt is sufficient to fill even the smallest structural details down to the submicrometer range. After complete mold filling, the tool containing the polymer has to be cooled until the strength of the molded part is sufficient to ensure defect-free demolding. Due to the high fragility of most microstructures, highly precise tool movements have to be guaranteed. For an automatic process conduct, the control software of the injection molding machines has to be backfitted in such a way that all additional functions of micro-molding are implemented in the automatically running sequence as well.

A comprehensive description of micro injection molding technology with a focus on powder processing can be found in [1].

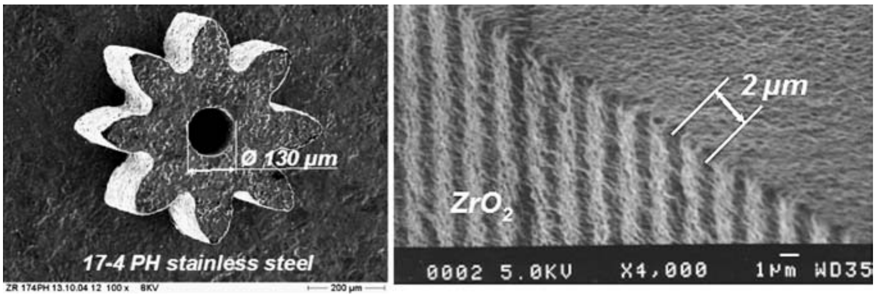
3. Micro powder injection molding

3.1. Micro powder injection molding (MicroPIM) in general

Micro injection molding using polymer materials has become well-established in industrial manufacturing; on the other hand, many applications require material properties (mechanical, thermal, abrasion) that cannot be met by polymers. From this point of view, extension of powder injection molding to the micro scale was quite obvious [2, 3].

At Forschungszentrum Karlsruhe micro powder injection molding was developed using powders and binder systems which have already been in service for macroscopic applications.

As typical test structures, gear wheels with 560 µm minimum diameter were manufactured using zirconia powder. The mold inserts were manufactured by the LIGA process (LIGA is the german abbreviation for the process sequence of lithography, galvanoforming and replication) which uses X-ray irradiation for the lithographic structuring step. Spectrometer test structures represent another kind of micro op-



(a) (b)

Fig. 1. Sintered LIGA test structures made by Powder Injection Molding. (a) Gear wheel in stainless steel 17-4PH, (b) Spectrometer test structure with a step height of approx. 0.8 µm in zirconium oxide.

tical example leading to the current limitations of MicroPIM: As shown in Fig. 1a and b, replication of structural details smaller than 1 µm is possible. However, the performance of the whole part is determined by the surface quality rather than by the structural geometry.

The lowest weights of the parts (LIGA gear wheels made of zirconia ceramic) after separation and finishing were just 0.25 mg.

The manufactured micro parts achieved a theoretical density of up to 97 % for metals and aluminum oxide, whereas the values for zirconium oxide reached 99 %. Due to the high surface-to-volume ratio of micro components, debindering and sintering are usually less crtitical procedures in micro manufacturing than in macroscopic fabrication. For example, the zirconia gear wheels mentioned above could be processed – without any optimization – by typical parameters, here a maximum sintering temperature of 1450 °C at a holding time of 90 min. Maximum heating/cooling rates were 5 K min^{-1}.

Linear shrinkage varied in a range of 15–22 % depending on the composition of the feedstocks. Due to the smaller particle size of the ceramic powders, the molded structures possess a better surface quality (R_a = 0.2 µm) compared to e. g. metal micro parts (R_a = 0.5–0.8 µm).

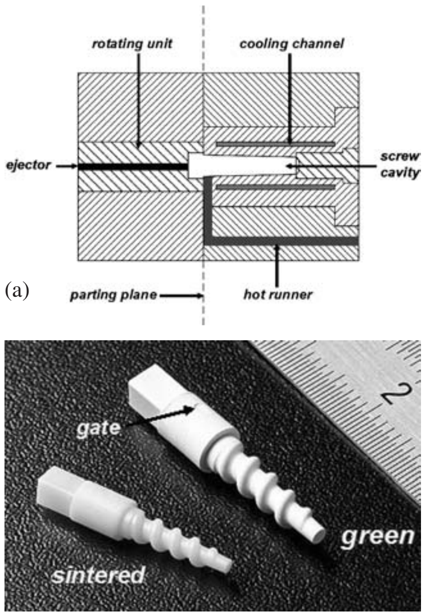
The experiments described above demonstrated that the particle size distribution has a significant influence on the accuracy as well as on the surface quality of the replicated structures: Best results have been achieved by using ceramic powders with a mean particle diameter of 0.5 µm or even smaller (see Table 1). Therefore, the demand to process finer powders even including the utilization of nano powders can be regarded as one of the most important results of the investigations carried out up to now.

To widen the application opportunities of MicroPIM attempts were started within the Collaborative Research Project SFB 499 to manufacture more complex shaped devices. As a demonstrator a nearly free-formed feed screw for a micro-dispenser unit was chosen. Production of the tool inserts was carried out by milling and electro discharge machining (EDM). For replication a modified micro injection molding process was introduced to PIM fabrication; usually, such screws are manufactured in tools containing two or more clamping units to release the cut-back geometry of the screw. However, the occurance of burrs can never be avoided completely, so a novel spindle construction was developed to unscrew the just molded green body before tool opening (Fig. 2a). Initial trials were carried out with zirconia feedstocks, and debindering and sintering could be carried out without major problems (Fig. 2b). At present, investigations on further miniaturization and on more complex sample geometries are underway.

3.2. Considerations on replication accuracy of the CIM process

3.2.1. Replication accuracies reached so far

Powder injection molding is a near net-shape manufacturing technology suited to mass production of complex



(a) (b)

Fig. 2. (a) Scheme of the new molding tool operating with screw-out demolding procedure, (b) Samples of screws as in the sintered state and as green bodies just after demolding by unscrewing and ejection.

Table 1. Technical data of micro powder injection molding as a function of the powder used.

Material	d_{50} (µm)	Max. aspect ratio	Min. structural detail (µm)	Density (% theo.)	R_{max} (µm)
Al_2O_3	0.4–0.6	>10	<20	97	3
ZrO_2	0.2–0.4	>10	<3	99	<3
for comparison:					
316L	4.5	10	50	97	8

shaped metal or ceramic parts with high quality and relatively tight tolerances. Especially for ceramics where the high hardness of the sintered parts requires expensive diamond tools for reworking, increasing the dimensional quality can reduce manufacturing costs remarkably.

The influence of process parameters on part dimensions and dimensional tolerances has been a key question in process control since the early days of PIM [4] and is still the subject of many research projects [5–7].

According to German, tolerances of 0.2 to 0.5 % can be achieved by PIM [8], while ±0.1 % is mentioned as a reference value for the minimal dimensional tolerance [9] possible with this technology. [10] refers to an example where a tolerance of ±0.05 % was obtained for some critical dimensions of injection molded ceramic parts for jet engines with a process yield of 80 %, whereas the actual process had a scatter of ±0.12 %.

Injection molding parameters obviously have a great effect on the dimensional quality of the final sintered part. According to German, molding parameters typically are the source of 60 % of the current process variation [10]. Nevertheless, the subsequent steps are very important, as well: during debinding, when the organic binder material is extracted, the remaining porous powder framework is extremely fragile and vulnerable, and parts are easily damaged. The biggest dimensional change in the process chain occurs during sintering where a linear shrinkage of about 20 % takes place. Hence, controlling debinding and sintering is also very important for part quality in PIM [11].

3.2.2. Studies on dimensional quality of cylindrical precision ceramic parts (ferrules)

Several studies on the dimensional tolerances of injection molded single-mode ferrules (see Fig. 3a and b) made of ZrO_2 have been performed to analyze the influence of process parameters on part quality in CIM [12–14]. These investigations were mainly part of the project "Micro-P-PIM" funded by the BMBF. A very precise cylindrical mold cavity (manufactured by Junghans microtec GmbH) with a diameter of 3.199 mm, a cylinder form tolerance of ±2.5 μm and a surface quality R_a of 0.24 μm was used. The samples were produced on a conventional injection molding machine Arburg Allrounder 420C 600-100 under laboratory conditions.

Injection molding pararameters were optimized for better dimensional tolerances and surface quality with design of experiments using the response surface method [14]. For the experiments, the commercially available zirconia feedstock Inmafeed® K1011 (Inmatec Technologies Ltd.) was used.

The optimized process parameters were tested in a long-term experiment producing parts for about eight hours with constant molding conditions. 890 ferrule green parts were molded. Quality control was performed on a sample of 250 green parts. 160 of these 250 ferrules were measured again after debinding and sintering.

Debinding of the green compacts was done in two stages: at first, the parts were placed in distilled water (25 °C) for about 20 h. After drying, thermal debinding and sintering at 1500 °C took place in a chamber furnace Carbolite RHF 17/3 E with the temperature program recommended by the feedstock producer.

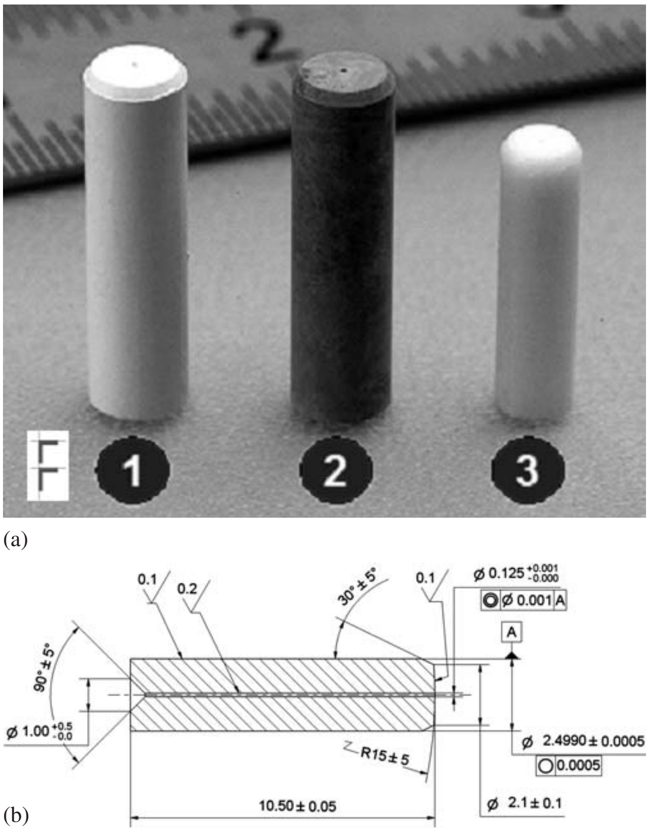


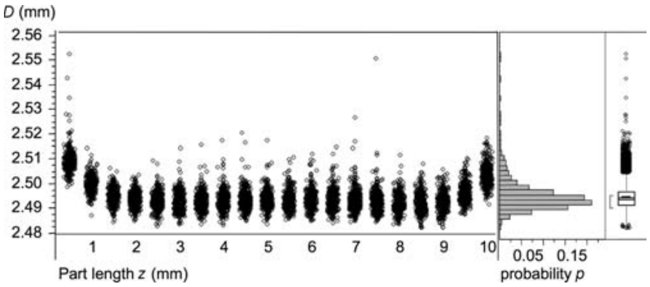
Fig. 3. (a) Stages of the CIM process: green compact (1), brown part (2) and sintered part (3) of single-mode ferrule, (b) product specification for single-mode ferrule (dimensions in mm).

Outer diameter D, length L and average roughness R_a of green and sintered parts were analyzed. The variation coefficient of diameter, length and roughness was used as a measure of process stability.

The outer diameter and roundness of the parts was measured in the green and sintered states in a contact-free manner at 80 points over length and perimeter. A specially designed measuring device based on a laser scanner Z-Mike 1210 Gold LX (repeatability ±0.15 μm) equipped with a high-precision rotational axis was used. The length of the ferrules was measured with a length gauge CT 60M (Heidenhain Ltd.). The surface quality was determined with a MicroGlider® (Fries Research Technology Ltd.) using a CWL optical sensor CHR 150N. The part weight was determined with an industrial scale Sartorius BP211D.

The results of the preliminary studies showed that the diameters of the green and sintered parts vary over part length and perimeter in a range of about 15 to 30 μm [13]. This variation is primarily due to the fact that the diameter of the parts is larger at the ends of the cylinder compared to the middle. A simulation of the mold filling and warpage of the molded part with the software Moldex3D showed the same typical part deformation [15]. Hence, it is assumed that this form tolerance is an effect of non-uniform shrinkage during cooling of the feedstock in the mold, as the ends of the cylindrical part are cooling faster than the middle creating a "micro sink mark" in the middle of the part.

The green and sintered parts produced in the long-term experiment showed the same typical form tolerance. Figure 4 displays the distribution of all measured diameter values of the 160 sintered ferrules. The distribution has a



(a) (b)

Fig. 4. Distribution of measured outer diameters (11510 values) of 160 sintered parts over (a) part length and (b) as a histogram with outlier box plot.

variation coefficient of 0.21 % (standard deviation 5.3 µm, mean 2.495 mm). The variability chart on the left shows the distribution of the values grouped by the measuring position in part length. It can be seen that a remarkable part of the dimensional variation is found at the ends of the part ($z < 2$ mm and $z > 9$ mm), whereas the dimensional scatter in the middle of the part is less pronounced. The histogram with the outlier box plot on the right represents the distribution of all measured diameter values. 99 % of them can be found within an interval of 29 µm which corresponds to a 3σ-tolerance of 1.16 % and the interquartile range is very narrow.

The length distribution of the 160 sintered ferrules is shown in Fig. 5a. The distribution has a variation coefficient of 0.2 % (standard deviation 19.9 µm, mean 10.143 mm).

Figure 5b displays the distribution of the average roughness values of 160 sintered ferrules. The best parts have a surface quality R_a of 0.26 µm while 90 % of the values are below 0.57 µm.

The preliminary studies showed that sintering shrinkage of the parts is not completely isotropic [13]. The long-term experiment led to the same result: while the diameter change from the green compact to the sintered part is about 21.2 %, shrinkage in length is only about 20.8 %.

Part quality decreased strongly from the green to the sintered parts: the average cylinder form tolerance doubled and the average of the roughness values increased by nearly 50 %.

4. Micro Two-component Powder Injection Molding (2C-MicroPIM)

4.1. Objectives of 2C-MicroPIM

Handling and mounting of micro components are rather complicated procedures. This is not only due to the tiny dimensions, but, additionally, to the risk of damaging and the sensitivity to contamination which rises with decreasing structural sizes.

A promising possibility to reduce this difficulty is to integrate the shaping and mounting procedures in one process step by multi-component injection molding. This technology is widely used in macroscopic fabrication on a sophisticated technical level. Due to the high economic efficiency further growth of application can be expected. From this point of view adapting multi-component technology for the fabrication of micro components, as well, is an interesting option.

Additionally, multi-component injection molding offers the possibility of connecting different materials within one part, thus combining different material properties. Conductive/insulating, hard/soft, or magnetic/non-magnetic combinations are only a small selection of a number of possibilities [16, 17].

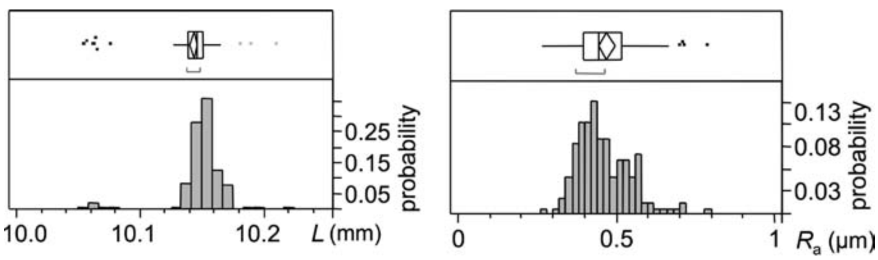
It has to be mentioned that due to the high attractiveness of 2C-PIM a few patent pendings have been granted in recent years covering particular versions of two or multi-component PIM-parts, e. g. in [18]. This constellation, of course, has to be considered in case of eventual practical applications.

Two-component micro injection molding is considered to be a viable process for the series production of micro components from multi-functional materials in the long term. Machinery and tooling equipment were developed at Forschungszentrum Karlsruhe and resulted finally in a worldwide unique combination of three special subvariants of injection molding: two-component micro powder injection molding (2C-MicroPIM).

In multi-component-PIM, (at least) two feedstocks filled with different powders are injected into a thermostated tool more or less simultaneously to produce a green compact from the compound [19]. These feedstocks used for two-component micro powder injection molding have various, partly opposite functions. Complete filling of microstructured mold areas will be ensured by their low viscosity, deformation-free demolding by high mechanical strength, pressure-free debindering by low swelling, and distortion-free sintering by homogeneous powder distribution in the green compacts and isotropic shrinkage. The objective is to produce a compound of high adhesion strength or defined moving operations against each other from at least two different ceramics [20].

4.2. Development of 2C-MicroPIM exemplified by demonstrator "Shaft-Gear Wheel Combination"

An interesting example encompasses the 2C molding of a shaft – gear-wheel combination. For realization, a new 2C-tool with a rotating index plate plus complete micro replication features had to be developed (Fig. 7a). Due to the



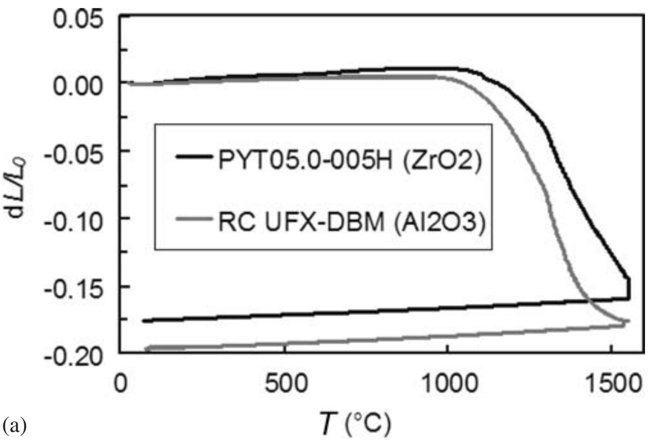
(a) (b)

Fig. 5. (a) Distribution of measured lengths of 160 sintered parts, (b) distribution of measured average roughness R_a of 160 sintered parts.

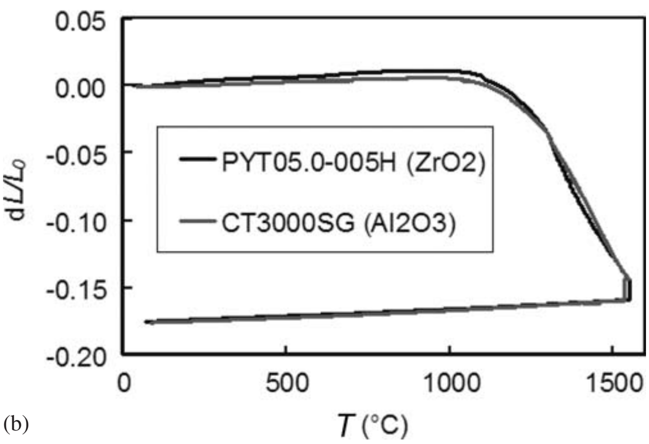
reduced shaft diameter in the bearing section hindering axial movement of the gear wheel, such a two-material component could not be produced by a simple mounting of two singular parts. Each subproduct can be molded using a particular material so that combinations of two ceramics, two metals or even metal and ceramic are in principle feasible within one injection molding cycle [21, 22].

The procedure in general can be configured to obtain both mobile and immobile bonds. With respect to mobility, the composition of the feedstocks and here especially the powder–binder ratio is of great importance: For obtaining a mobile junction, it is advantageous if the core section starts to shrink before the outer section does and the shrinkage rate of the core has to be higher than that of the outer section. For realizing an immobile connection the outer section must show a slightly higher shrinkage rate and an earlier start of sintering than the core section. It has to be mentioned that the sintering behavior has to be adjusted well to avoid any kinds of cracks or to achieve a good movability, respectively. Luckily, the sintering activity of the powders depends mainly on the mean particle size so that shrinkage rate and sintering start temperature can be varied by the right choice of powders.

For this purpose, using a dilatometer can be very advantageous. Figure 6a and b show dilatometer curves of examples for a possible combination of Al_2O_3 and ZrO_2 powders for realizing an immobile junction as well as a possible combination for realizing a mobile junction.

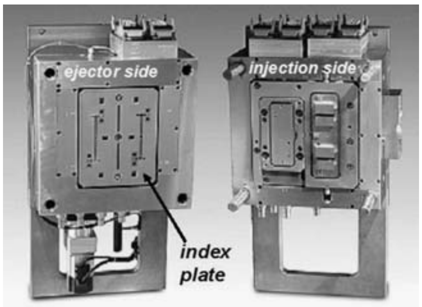


(a)

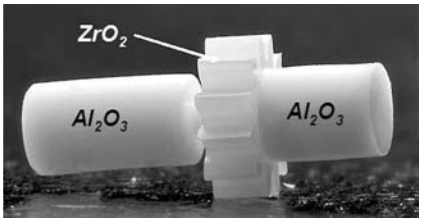


(b)

Fig. 6. Relative length changes of selected ZrO_2/Al_2O_3 powders during sintering. (a) combination promising for mobile bonds, (b) possible combination for an immobile bond.



(a)



(b)

Fig. 7. 2C-Micro injection molding of shaft–gear-wheel combinations. (a) New 2C-tool with turnable index plate, (b) two-component zirconia/alumina sample.

Experiments started with the aim of realizing immobile bonds using a combination of alumina as shaft material and zirconia for the gear wheel. Using feedstocks with adjusted powder contents (approximately 55 vol.%) and a carefully designed sintering program, tight and stable connections were achieved despite the fact that neither ceramic usually shows a considerable thermodynamic miscibility (see Fig. 7b).

At present, trials to obtain mobile connections are being carried out. Additionally, there are experiments using metal feedstocks, i. e. filled with fine grain 17-4PH steel powders. Although steel combinations have already been tested for immobile and mobile bonds of macroscopic samples [23], transfer to micro technology results in additional challenges: Due to the very small distances between the two green/brown portions it is quite difficult to prevent them from reconnecting during sintering so that, e. g., novel sinter bearing solutions have to be developed.

5. Outlook

Not least due to its high technical and economical potential, development of micro replication of ceramic materials will not remain at the current stage and further progress driven by the requirements of industrial and scientific customers can clearly be expected.

In order to enhance the performance capability of MicroPIM, further materials and process development is going on. These experiments will deal mainly with the utilization of very fine, under certain conditions even nano powders, for micro specific feedstocks.

As powder injection molding is a quite complex process dimensional control for production of precision parts with PIM is a difficult challenge.

Currently, further studies on the influence of process parameters on the quality of single-mode ferrules in ceramic injection molding are in progress to improve knowledge about the process and its effects on precision parts. Furthermore, attempts to generate a novel tool concept and improve the way of process operating enabling the deliberate manipulation of molding and cooling procedures are currently under consideration.

Computation and simulation might help to reduce corrections of tool dimensions to obtain the desired dimensions, especially if extreme precision is required. An important prerequisite is, of course, the availability of novel simulation routines suitable for PIM.

Entirely new prospects are opened up by multi-component micro injection molding. A promising technological level has been reached but there is, of course, a lot of research work to be done, especially concerning the adjustment of feedstock formulations and the optimization of sintering procedures. 2C-MicroPIM will not only result in reduced assembly expenditure for micro systems, but also in the production of new functional units. Therefore, it offers a clear economic and technical potential for future applications.

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