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Ceramic Injection Moulding using 3D-Printed Mould Inserts

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In general, micromilled mould inserts made of steel, aluminum or brass are used today for ceramic injection moulding (CIM) processes. However, tool making via mechanical subtractive manufacturing processes as micromilling is time- and cost-effective and the use of 3D printed mould inserts becomes an attractive alternative to metal mould inserts. In this paper, we report about the use of 3D printed mould inserts for CIM of alumina microreactor parts. It was observed that mould inserts printed using the Polyjet technology were very well suited for functional prototyping via CIM. The mould inserts surface was found without visible thermally introduced damage after twenty CIM process cycles. In contrast to the high quality of mould inserts printed using the PolyJet technology, mould inserts made via fused deposition modeling (FDM) technology revealed as not applicable for the purpose of this study. The mould inserts manufactured using FDM-printer exhibit significantly higher surface roughness, larger longitudinal deflection and manufacturing-related undercuts along the edges of the 3D printed microstructure.

Keywords: Ceramic Injection Moulding (CIM), 3D Printed Mould Inserts, PolyJet, FDM, Rapid Tooling.

1. Introduction

The Ceramic Injection Moulding process (CIM) is one of the essential replication processes for mass production of functional ceramic components. In general, the moulds used for CIM processes are fabricated from steel, brass or aluminium using milling processes. Subtractive manufacturing such as the milling process fits the bill in high volume production. In case of design studies, milling of metal mould inserts for each single design relatively time-consuming and therfore also cost-intensive.
Faster tooling will offer a new freedom in rapid prototyping and allow systematic design studies and digital fabrication for customized items starting for lot size of one and Additive Manufacturing (AM) is becoming a viable option for the production of injection moulding inserts in pilot production settings. [1,2].
Actually, the combination of 3D Printing (3DP) and CIM technology offers a highly attractive approach to realize a variety of differently structured ceramic parts in a short time and particularly in small batch-production and rapid prototyping applications the use of 3D Printing (3DP) as a rapid tooling technology is gaining importance in the last three years.
The first 3D printed moulds were fabricated and very successfully used for manufacturing of non-planar micro- or millifluidic chips or even elastomeric dielectric layer in pressure sensors by casting them from PDMS [3,4,5]. In 2015, Mohanty *et al.* actually used water dissolvable 3D printed moulds with different infill patterns from PLA as sacrificial moulds to cast PDMS around them and fabricate complex scaffolds [6].
Last year, 3D printed moulds were successfully tested for plastic injection moulding and hot embossing processes where they have to withstand significant higher temperature and pressure [2,7,8]. In June this year, Altaf *et al.* published their work about a comparison of the performance of 3D printed polymer moulds with an aluminum mould for potential use in Metal Injection Moulding (MIM) process and reported that 3D printed moulds could successfully be used for a limited number of MIM cycles where the design is subjected to rigorous testing and iteration before finalization [9].
In this study, 3D printed mould inserts have been tested for CIM of alumina feedstock. Therefore, the thermoplastic alumina compound is heated within a heated screw conveyor to form a plastified melt which can be easily injected under pressure through an axial feed into the cavities of a closed 3D printed mould. During the injection step, a pressure which is counteracted by the clamping unit of the injection moulding machine is built up and maintained until the injection moulded material has solidified. Then, the mould opens and the shaped ceramic green body is ejected. The CIM process may therefore be devided into four steps: Plastification, injection, holding, and ejection [8].
Due to the very low heat conductivities of plastic moulds in contrast to metal moulds it has to be considered that for CIM processes with 3D printed moulds the holding time has to be slightly extended since the to injection moulded mass will need more time to solidify. However, this is necessary to make the ceramic green body formstable enough for the ejection step.
During the injection moulding process the printed material has to withstand the contact to the hot moulding compound (100 - 200 °C) and also high injection pressures above 20 MPa. For choosing the right 3D printable material and technology for rapid tooling, factors like material compatibility, surface finish and part size are to consider. The different 3DP technologies and materials have special characteristics and benefits. But for

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mould inserts, particularly the surface finish and resistance is of importance. Thus, it ensures an unconstrained de-moulding process which is crucial for avoiding deflections or damage in the injection moulded ceramic green bodies.

2. Materials and Methods

2.1. Design of mould inserts

Testing 3D printed mould inserts for CIM processes was performed in course of the development of a new ceramic microreactor with a special sensor system for electrical in-situ monitoring of continuous hydrothermal syntheses (CHTS) of metal oxide nanoparticles [10]. Beside of mould inserts milled from brass, different 3D printed mould inserts were used to find out if the PolyJet or the Fused Depositon Modeling (FDM) technology is more suitable for manufacturing mould inserts for CIM processes. Both 3DP technologies were used to manufacture mould inserts with the design of a microreactor for CHTS, depicted in Figure 1. It comprises the channel structure of one microreactor half. The benefits of the 3DP technologies have been compared with respect to the quality of the microreactors green bodies.

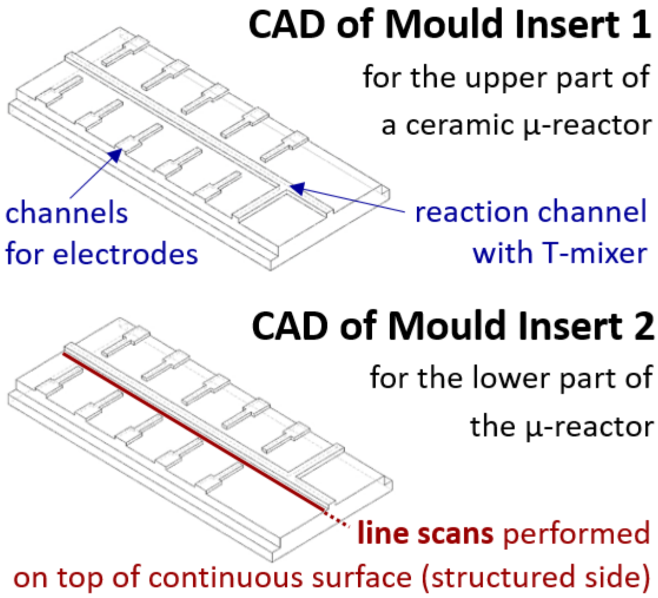

Figure 1. CAD of mould inserts for an injection moulding process of two ceramic microreactor halves. The red line indicates the course of the surface contour screening.

2.2. Materials for mould inserts

Mould inserts have to meet certain requirements. They should combine a good chemical surface stability with a high dimensional accuracy and a roughness in the sub-micrometer area for both, the microstructures as well as the continuous surfaces. Furthermore, the ideal mould insert exhibit a high degree of hardness, mechanical durability and a good heat conductivity to be capable of quickly dissipating the thermal energy after each injection step. Some chosen thermal and mechanical properties of the 3D printed materials used in this study have been found in literature and are summerized in Table 1. In addition, the heat conductivity of the PolyJet material VeroClear RGD 810 was determined also for increased temperatures by measuring the thermal diffusivity using Laser Flash Analysis (LFA 427, NETZSCH) and the specific heat capacity using Differential Scanning Calorimetry (DSC 204, NETZSCH) on a 1 mm thick sample that was cut out from a mould insert. The values are given in Table 2.

Table 1. Thermal and mechanical properties of polymers used for 3D printing of mould inserts.

Polymer	ABS	PEEK 450	VeroClear RGD 810	Digital ABS
Glass transition T_g °C	103 [11]	147 [14]	52 – 56 [16]	47 – 53 [16]
Shore hardness Scale D	75 [12]	84 [14]	83 – 86 [16]	85 – 87 [16]
Modulus of elasticity GPa	1.7 [11]	3.7 [14]	2.0 - 3.0 [16]	1.7 - 2.2 [16]
Heat conductivity (20 °C) $Wm^{-1}K^{-1}$	0.1 [13]	0.25 [15]	-	-

The PolyJet materials have a markable hardness, comparable to PEEK. However, ABS is significantly softer and also its modulus of elasticity is the lowest one in comparison to the other printed materials. This is unfavourable for the use as mould in a CIM process since high pressures are applied and the mould should withstand them. In contrast, the FDM materials exhibit significant higher glass transition temperatures and seems to be thermally more stable against the hot moulding compound that is injected. Particularly, PEEK with a T_g above 146 °C and also an acceptable heat conductivity (which is important for a fast heat dissipation after the injection) seems to be a promising material for mould production.

Table 2. Injection moulding parameters.

Injection temperature	°C	160
Injection pressure	bar	286
Injection speed	$mm \cdot s^{-1}$	100
Injection time	s	0.57
Holding pressure	bar	210
Holding time	s	2
Tool temperature	°C	30

2.3. 3D printing of mould inserts

Decisive for the application in CIM processes is not only the thermal and chemical stability of the mould but also the printing quality and especially the surface finish. For CIM of ceramic microreactor parts, 3D printed mould inserts for each microreactors half have been manufactured from different materials depending on the 3D printing technology. By means of a line scan along the longitudinal axis of each sample, depicted in Figure 1, the mould inserts were characterized using the white-light interferometer MicroProf® (FRT GmbH, Germany). The total deflection was determined as well as the roughness accordingly to DIN EN ISO 25178 to analyse the surface finish of the mould inserts. The 3D printed mould inserts are depicted in Figure 2 to Figure 5.

2.3.1. Via PolyJet with Digital ABS Green

Mould inserts printed by a PolyJet printer have been manufactured using the PolyJet printer Objet 260 Connex 3 (Stratasys GmbH, Germany) that allows the use of 1 up to 3 polymers and a support material within one print. The maximum build size of the printer is 255 x 252 x 200 mm³. The printed material Digital ABS Green (Stratasys GmbH, Germany) is a multi-material photopolymer

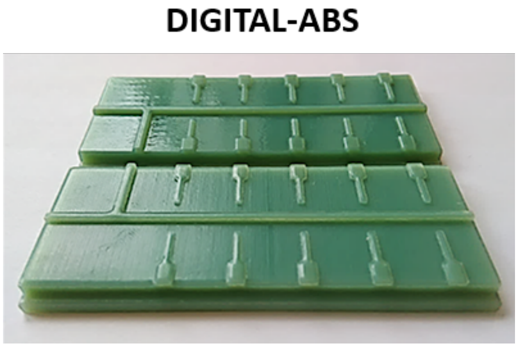

Figure 2. 3D printed mould inserts made of the multi-material Digital-ABS Green.

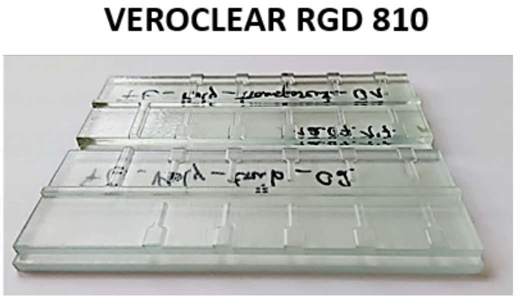

Figure 3. 3D printed mould inserts made of the transparent polymer VeroClear RGD 810.

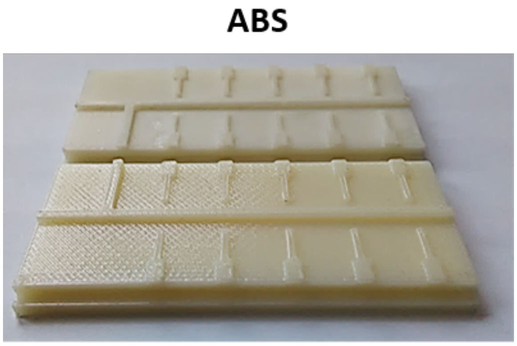

Figure 4. 3D printed mould inserts made of ABS.

Figure 5. 3D printed mould inserts made of PEEK.

(thermoset) and due to its core shell material combination of two polymer components (RGD515 and RGD535) this composite material has the highest impact resistence (90-110 J/m) [16] of all PolyJet materials. Though, its heat deflection temperature (HDT) lies according to manufacturer only between 58 and 68 °C.
The parameters within the printing process itself are already optimized by the manufacturer of the printer and only some minor settings can be adjusted. For example, there is the printing option "glossy or matt" that defines where support material will be used and it can result in different surface roughnesses and in varying surface appearance. In this course, the option "matt" was used since this results in more uniformly high surfaces up to the edges, while the glossy setting would smoothen the contours and edges. It would look shiny but the printed parts would also lose structural details. The layers are processed with a thickness of 30 μm resulting in a resolution in z-direction of 800 dpi. In x and y direction within the layer, the resolution for each is 600 dpi.

2.3.2. Via PolyJet with VeroClear RGD 810

Using the same PolyJet printer as in 2.1.3 mould inserts made of the single polymer material VeroClear RGD 810 (Stratasys GmbH, Germany) were also printed. The printing parameters were retained, only the layer thickness in the single material mode was reduced to 16 μm resulting in a resolution of 1600 dpi.

2.3.3. Via FDM with ABS

The FDM printer used within this research was a X350 pro (German RepRap GmbH, Germany) with the slicer program Simplify3D. Mould inserts were printed from a commercial acrylonitrile butadiene styrene filament (ABS natur, German RepRap GmbH, Germany) with a layer thickness of 0.1 mm. According to the fine layer thickness the nozzle diameter was 0.25 mm. ABS is a thermoplastic standard polymer with a glass transition temperature in the range around 105 °C. It was printed with an infill of 95% and a wall thickness of three lines in outside-in direction for best geometrical precision. The printing and heat bed temperature was set to 235 and 110 °C.

2.3.4. Via FDM with PEEK

Mould inserts made from polyetheretherketone (PEEK 450 Natural, Apium Additive Technologies GmbH) were printed commercially at Apium Additive Technologies GmbH also by means of an Apium FDM printer. PEEK is a semi-crystalline thermoplast. Due to its high glass transition temperature T_g of 146 °C and a melting temperature Tm of 338 °C, it is stable over a wide temperature range (from -196 °C to 260 °C) [13]. The combination with its chemical and wear resistance makes PEEK a high performance polymer that is suitable for application in injection moulding processes.

2.4. CIM of alumina microreactors

For performing the CIM process, the 3D printed mould inserts have been installed into an injection moulding machine (ELECTRA S 50, Ferromatik Milacron GmbH, Germany) as shown in Figure 6. The ceramic moulding compound to be injection moulded has been prepared by mixing the sub-micron-sized alumina powder Martoxid® MR70 (99.8% Al_2O_3 with 0.08 wt.-% SiO_2 and 0.06 wt.-% MgO addition, Martinswerk®, Germany) with an average particle size of d_{50} = 0.5 - 0.8 μm (d_{90} < 3 μm) and a specific surface of A_{sp} = 8.6 m^2/g with several organic additives for 1h with 30 rpm at 125 °C applying a mixing kneader (Plastgraph, Brabender GmbH). As dispersant stearic acid (≥ 98 %, Carl Roth GmbH, Germany) was used.
The binder system comprises a polyvinyl butyral (PVB) powder (Mowital B30 H, Kuraray Europe GmbH) as the back-bone polymer and a short-chain polyethylene glycol (PEG 4000, Rotipuran® Ph.Eur., Carl Roth GmbH) that acts also as a plasticizer. The moulding compound was heated up to 160 °C within the screw conveyor of the injection moulding machine before injection. At this temperature the moulding compound was plasticized enough to be injected with a pressure of 286 bar into the mould. This corresponds to an injection speed of 100 mm/s.
Further process parameters are listed in Table 2. To prevent distortion of the patterned ceramic green bodies they were cooled within the moulding tool. The tool temperature was set to 30 °C to prevent the plastic mould inserts getting too hot.

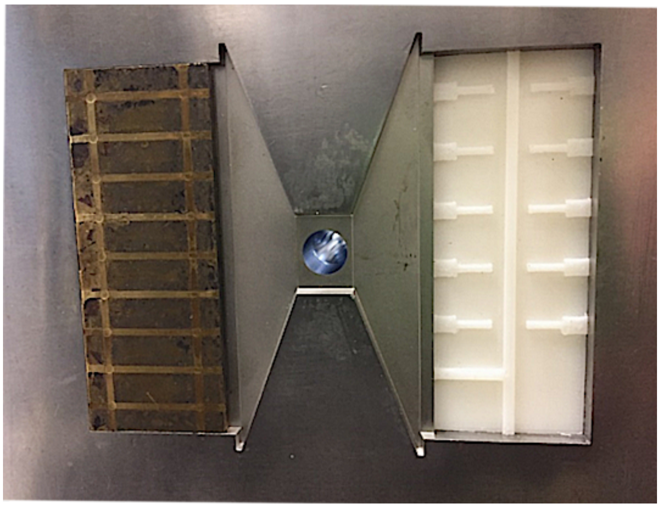

Figure 6. 3D printed mould insert (ABS) installed into the injection moulding machine for performing the CIM process.

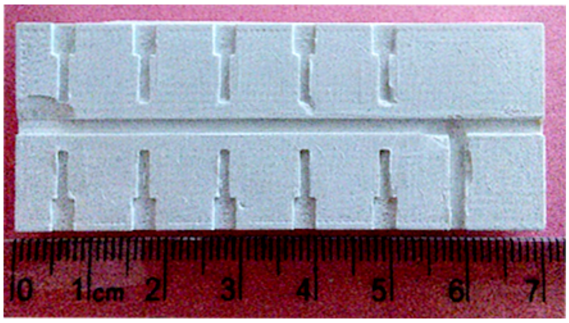

Figure 7. Structured green body manufactured via injection moulding using an ABS mould insert.

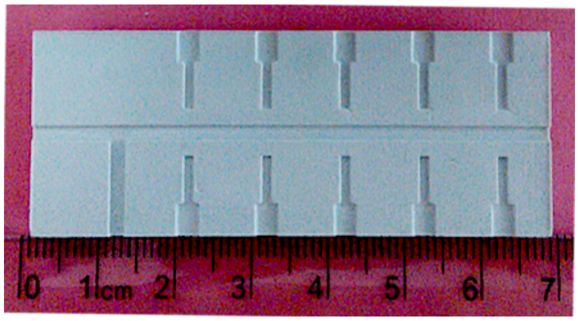

Figure 8. Structured green body manufactured via injection moulding using a 3D printed mould insert made of VeroClear RGD 810.

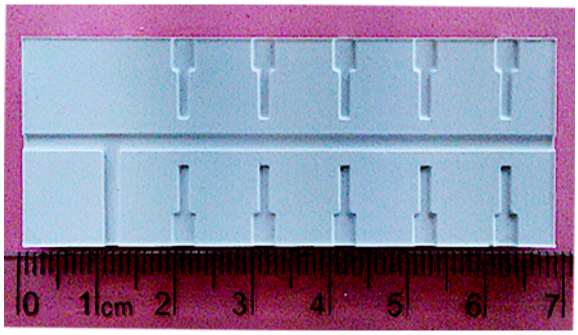

Figure 9. Structured green body manufactured via injection moulding using a 3D printed mould insert made of Digital-ABS.

3. Results

3.1. Visual inspection of the mould inserts' structural accuracy and their suitability for CIM

Comparing the pictures of the mould inserts, shown in Figure 2, Figure 3, Figure 4 and Figure 5, it can be seen that the structural accuracy and the surface finish of the 3D printed mould inserts depend on the printing technology.

While the PolyJet printed ones show an excellent surface finish and a very high and uniform structural accuracy, the FDM printed mould inserts are of lower quality. Their single bars of the mould inserts structure (for forming reaction and electrode channels) show on visual base dimensional inhomogenities in all directions. Their highest layers form an overhang at their edges which acts like an undercut when the replicated part is demoulded. These undercuts hinder the demoulding process significantly and lead to abruptions in the moulded part along the edges of the microstructure. Figure 7 shows a ceramic green body using the FDM printed mould inserts made of ABS. Here, the reported defects along the edges of the structure in the moulded part are clearly visible. These defects are caused by a flattened front side of the micro-structure on top of the mould insert. Such defects are technically unacceptable.

Both, the ABS as well as the PEEK mould inserts had not the required quality with respect to their structural accuracy to work well at demoulding. However, the demoulding success is a crucial point in the production chain of the CIM technology. Additionally, the ABS mould inserts was thermally not stable. Despite the low tool temperature of 30 °C, the structured surface was heated up that much by the moulding compound that already after 3 to 5 injection cycles there were streaks visible on the surface made of melted ABS. For the production of mould inserts the FDM technology did not provide pleasing results. The mould inserts 3D printed in course of this study from PEEK, lack dimensional accuracy that much that they were not even used for moulding. However, although their surface finish was not suited for technically acceptable moulding, the PEEK mould inserts show a good rigidity which is correlated to its high modulus of elasticity (Tab. 1).

In contrast to the via FDM printed mould inserts, the mould inserts printed via PolyJet, shown in Figure 2 and Figure 3, maintained their excellent surface finish and the smooth edges along the structure also after their use in the CIM process. The CIM process could be performed easily with these mould inserts and also demoulding of the injection moulded parts took place automatically without any visible defects in the ceramic green bodies. The 3D printed materials VeroClear RGD 810 and Digital ABS green resisted the thermal and mechanical stress during the CIM process and worked well for more than twenty CIM cycles. The patterned ceramic green bodies that were fabricated using the PolyJet mould inserts are shown in Figure 8 and Figure 9. However, if there is the choice between these two PolyJet materials, VeroClear RGD 810 seems to be the better choice. Its slightly higher modulus of elasticity (Tab. 1) seems to be an important benefit with regard to its application as a mould for injection moulding processes.

3.2. Thermal analysis of VeroClear RGD 810

Due to the particularly good suitability for CIM processes that the mould inserts made of VeroClear RGD 810 revealed, its thermal properties at increased temperatures are of interest.

The thermal diffusivity, the specific heat capacity and the heat conductivity are shown in Table 3 as results of DSC and LFA measurements. The density of the 3D printed mould insert from VeroClear was determined to 6.99 g/cm^3. Although, the thermal analysis shows that the PolyJet polymer VeroClear RGD 810 has

Table 3. Thermal properties of VeroClear RGD 810.

Temperature °C	Thermal diffusivity cm^2s^{-1}	Average spec. heat capacity $Jg^{-1}K^{-1}$	Heat conductivity $Wm^{-1}K^{-1}$
20	0.00107	1.337	0.100
50	0.00097	1.578	0.107
100	0.00094	1.905	0.125
150	0.00081	-	-

the same low heat conductivity as the FDM polymer ABS (Tab. 1), the CIM process could be performed well using the VeroClear RGD 810 mould inserts.

3.3. Characterization of the mould inserts' longitudinal deflection and surface roughness

The optical (white-light interferometer) and tactile surface inspection of the mould inserts´ topography reveals that all 3D printed mould inserts are not perfectly even but exhibit deflections of partially several hundred microns along their longitudinal axis. Figure 10 shows the deflections of each longitudinal line scan as measured. In Figure 11 the results of the topographical characterization are summerized to facilitate the comparison of the mould inserts´ quality. In contrast to the concave deflection of the mould inserts made of PEEK, VeroClear RDG 810 or Digital-ABS, the mould insert made of ABS has a convex deflection. However, the direction of the deflection does not matter but the total amount of the non-planarity. The more the mould insert is deflected, the more it has to be bent to be installed tightly into the tool holder of the injection moulding machine.

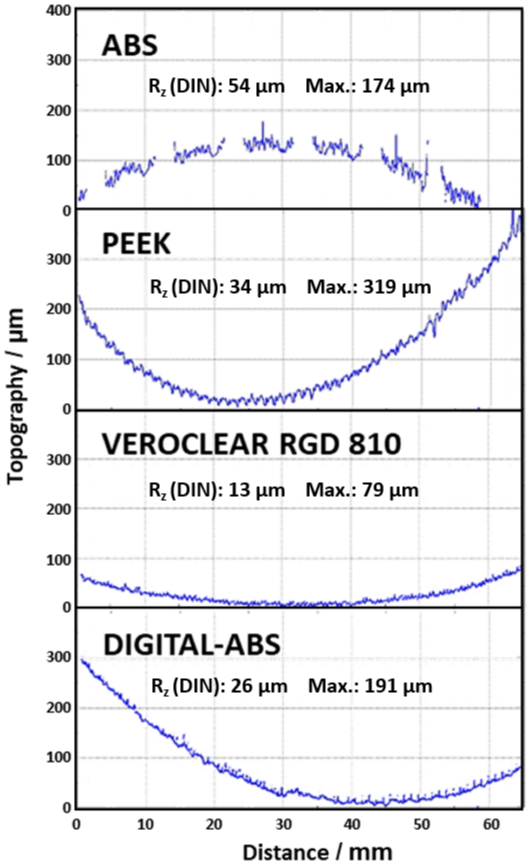

Figure 10. Line scans on the structured surface of different 3D printed mould inserts detected using white-light-interferometer.

Comparing the deflection of the ABS mould insert (printed with FDM) with the deflection of the mould insert made of Digital-ABS (printed with PolyJet) shows that the total longitudinal deflection is within the same range for both, the mould inserts 3D printed with FDM as well as with PolyJet. Both technologies did not provide perfectly planar tools. However, the surface of the PolyJet printed mould inserts is obviously smoother than of the FDM printed ones. The lowest surface roughness (R_{max}) with only 15 µm was measured for the mould insert made of VeroClear RGD 810. This sample showed not only the smoothest surface finish but also the smallest and additional most symmetrically distributed total deflection of only 79 µm which can be easily be taken up by installing the mould insert into the die plate of the injection moulding machine. Using the PolyJet technology in combination with this transparent material for 3D printing of structured tools achieved the best results with respect to a smooth and even mould inserts surface. Additional, the transparency of 3D printed tools can be an interesting feature since it opens up the possibility to observe visually the filling of the mould with the moulding compound while the injection process is performed.

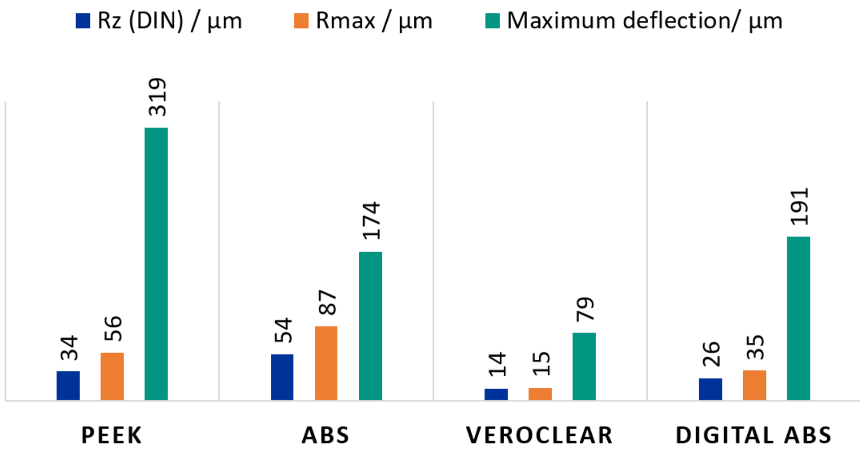

Figure 11. Maximum longitudinal deflection and roughness values of a line scan on top of the structured surface of the 3D printed mould inserts before use in the injection moulding process.

3.4. Quality of ceramic green bodies

As already reported in section 3.1, the quality of the FDM printed mould inserts (Figure 4 and Figure 5) was not applicable for the CIM process. Figure 9 shows a ABS mould insert with the ceramic green body on top of it that could not be demoulded neither automatically by the injection moulding machine as it should be performed in course of one injection moulding cycle nor manually afterwards. Due to the proceeded shrinkage of the green ceramic part while cooling down from the injection temperature (160 °C) to room temperature, demoulding is constrained additionally and when room temperature is reached, demoulding cannot be performed anymore without damaging the ceramic green body. Though, not all trials of demoulding from the ABS mould inserts failed, no defect-free green bodies could be obtained. The defects that partially occurred when the ceramic parts were demoulded from the ABS mould insert are shown in Figure 7 and Figure 12. Damages of the mould insert like liftings or detachments of single bars of the are depicted in Figure 12. They show what forces take place at the edges of the structure through canting of the materials

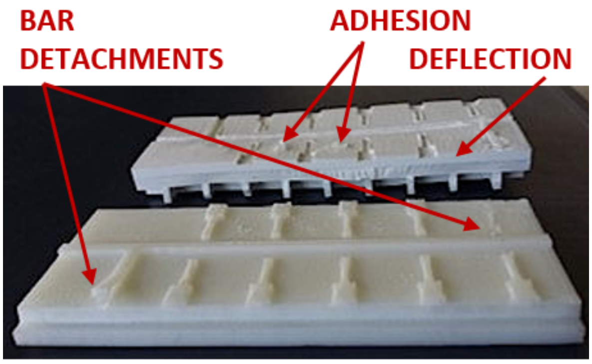

Figure 12. Defects like deflection of the green body and damages of its surface through a too strong adhesion to the mould insert as well as detachments of bars on top of the mould inserts structure.

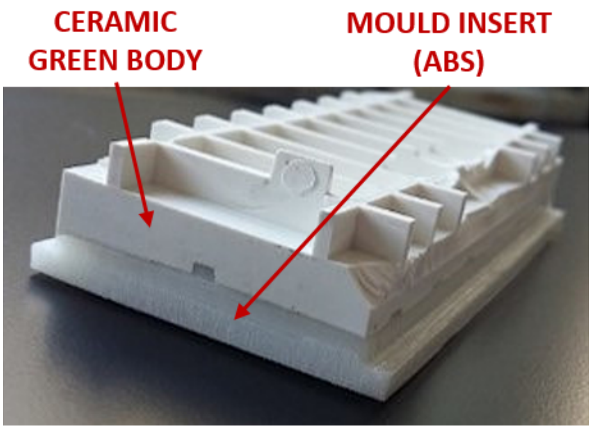

Figure 13. Failed demoulding process: The ceramic green body was not ejected automatically after injection of the moulding compound and remained manually not detachably bound to the mould insert.

while the demoulding process. The results of this canting are shown in Figure 7 as abruptions along the edges of the structure. Additionally, it can be observed from Figure 12 that adhesions between mould insert and ceramic part lead to damages on the surface of the moulded part. That the surface of the green bodies adopted the surface pattern of the 3D printed mould inserts is visible in Figure 7.

The green bodies surface showed the same patterns on their surface as the mould insert shown in Figure 4. In general, it was ascertained that the quality of the green bodies moulded using FDM printed mould inserts lays clearly below the quality of the green bodies moulded using the PolyJet printed mould inserts in terms of the structural accuracy. From the pictures in Figure 12 and Figure 13 can be observed that also the quality of the green bodies moulded with PolyJet mould inserts is superior in contrast to the ones moulded with FDM mould inserts. These injection moulded parts exhibit smooth surfaces, sharp edges and any visible defects.

Performing the CIM process using the PolyJet mould inserts provided technically acceptable green bodies that could be further processed very well. The only shortcoming was that they were also slightly deflected after demoulding but through a thermal post-treatment (1h @ 150 °C) these deflections could be easily remedied before stacking the moulded reactor parts together for further processing.

4. Conclusion

In a preliminary study 3D printed mould inserts made of ABS were used successfully for plastic injection moulding of polyoxymethylene (POM) and also polyethylene (PE). This gave rise to test the applicability of 3D printed mould inserts for CIM processes using ceramic feedstocks instead of commodity plastics. To take the higher operating temperatures into account that are commoly used for CIM processes, mould inserts were 3D printed via FDM not only from ABS but also from PEEK which is known for its high heat deflection temperature far above 200 °C.

Additionally, mould inserts were 3D printed using PolyJet technology to compare their applicability for the CIM process. It turned out that the quality of the moulds printed in course of this project via FDM was not sufficient to manufacture acceptable green bodies. The surface accuracy of the mould inserts was bad due to the high surface roughness (R_{max}) and undercuts along the edges of the microstructure.

This impeded the demoulding process and caused abruptions on their structured side of the injection moulded parts. The good part yield was restricted and a stable process could not be achieved using the mould inserts that were 3D printed via FDM. In contrast, the mould inserts that were 3D printed via PolyJet printer worked very well for CIM of alumina microreactor halves. The mould inserts made of the transparent polymer VeroClear RDG 810 exhibited with only 15 µm the lowest surface roughness and demoulding could be performed automatically without constraints.

Also the mould inserts made of the composite material Digital-ABS with a maximum surface roughness of 35 µm worked very well and defect-free green bodies with a high structural accuracy could be injection moulded using the PolyJet mould inserts.

It turned out that in our case the via PolyJet 3D printed mould inserts offer an attractive and applicable alternative to conventional metal mould inserts for prototyping and small production series of at least twenty injection moulding cycles.

3D printing of mould inserts via PolyJet provides several advantages in comparison with FDM. The parameters within the printing process itself are already optimized by the manufacturer of the printer and only some minor settings can be adjusted compared to a standard FDM-printer, where multiple settings and optimization processes needs to be taken into account.

PolyJet offers a better resolution in z-direction (16 µm compared to ~160 µm), very smooth and detailed and an almost non-porous printed solid part. The accuracy as well as the uniformity of printed details are significantly higher (deviations approximately in the range of 100 µm to 200 µm depending on the specific geometry, printer settings, material selection and orientation of the part on the building tray). PolyJet offers the better surface finish with a lower roughness and smoother contours and edges without losing details of structure and has the ability to create parts with great intricacy.

The main drawback of the PolyJet technology are the high acquistition as well as current costs (maintenance, consumables). While FDM printer can be obtained from 500 € (Miebmiebs HP) on, good PolyJet printer still costs at least 30 k€. In comparison to conventional metal moulds, the material costs for PolyJet polymers like VeroClear RGD 810 are higher (0.20 € for one mould insert made of brass with the here presented shape and 1.68 € for the same mould insert made of VeroClear RGD810). But taking into account that using 3D printing for toolmaking offers the possibility to fabricate ten or even more differently structured moulds in parallel within less than two hours, shows the high potential of AM in saving plenty of machining time. Despite the very low heat conductivity of plastic moulds in contrast to metal moulds formstable ceramic green bodies could be injection moulded with holding times of only two seconds. Hence, the cycle times are not very much longer.

Which mould making technology is to prefer, must be decided on a case-by-case basis. However, if only a few ceramic parts are required and a variety of mould designs shall be rapidly available, PolyJet printing has a great potential as rapid tooling technology for cost-effective design studies.

Acknowledgement

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