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Ceramic microfabrication by rapid prototyping process chains

R KNITTER and W BAUER

Forschungszentrum Karlsruhe GmbH, Institute for Materials Research III,
Postfach 3640, D-76021 Karlsruhe, Germany
e-mail: regina.knitter@imf.fzk.de

Abstract. Fabrication of micropatterned ceramics or ceramic microparts make high demands on the precision and resolution of the moulding process. As finishing of miniaturised or micropatterned ceramic components is nearly impossible, shaping has to be done by a replication step in the green, unfired state. To avoid high tooling costs in product development, a rapid prototyping process chain has been established that enables rapid manufacturing of ceramic microcomponents from functional models to small lot series within a short time. This process chain combines the fast and inexpensive supply of master models by rapid prototyping with accurate and flexible ceramic manufacturing by low-pressure injection moulding. Besides proper feedstock preparation and sufficient small grain size, the quality of the final components is mainly influenced by the quality of the master model. Hence, the rapid prototyping method must be carefully selected to meet the requirements of the component to be fabricated.

Keywords. Ceramic injection moulding; microcomponents; rapid manufacturing; rapid prototyping; microtechnology.

1. Introduction

In the last decades the development of microfabrication techniques has led to a miniaturisation of a diversity of mechanical, optical and fluidic components and systems. As almost all of the patterning techniques are appropriate only for selected materials, as a consequence, material available for microsystems technology is inevitably limited. Examples are the LIGA technique (German acronym for X-ray deep lithography, electrodeposition, and polymer moulding) for the production of microparts of polymers and selected metals, silicon technologies, micromachining of metals or patterning of UV-sensitive glasses. For some applications of microsystems, however, the specific properties of ceramics, like their high hardness, high thermal and chemical resistance or special piezo- or dielectric properties are of great significance. Unfortunately, just the first indicated properties make the use of the established micropatterning techniques impossible or not frugal for ceramic materials. As a result, the development and application of ceramic microcomponents have been retarded.

Ceramic microparts, like almost all ceramic components, are mostly formed in the green, unfired state by consolidating the ceramic powder with the help of more or less organic additives into the desired shape. In the past, a variety of processes were developed or known techniques adapted to the special requirements of the shaping of ceramic components with patterning details in the submillimetre range. These techniques differ in design restrictions, with respect to the attainable aspect ratio and the economic lot size, but have in common that a micropatterned mould is needed (Knitter *et al* 1996; Bauer *et al* 1997, 1999; Chan *et al* 2000; Piotter *et al* 2003). As most of these techniques require metallic moulds at least in one step of the replication process due to the applied pressure and/or temperature, these metal moulds are mainly produced by more or less expensive and time-consuming techniques like erosion methods, mechanical micromachining, or even the LIGA technique.

For the accelerated supply of models and prototypes, a large number of rapid prototyping (RP) methods have been developed in the last 15 years. Starting in 1986 with the stereolithography (SLA) of polymers (Hull 1986), the resolution and precision of RP techniques have been improved down to a few micrometres (Reinhardt & Götzen 1999; Bertsch *et al* 2000; Varadan *et al* 2001), and the variety of processes and processed materials has been expanded in recent years.

However, solid freeform fabrication techniques like multijet solidification (MJS) (Greulich *et al* 1995), selective laser sintering (SLS) (Subramanian *et al* 1995), laminated object manufacturing (LOM) (Klosterman *et al* 1998) or fused deposition of ceramics (FDC) (Bandyopadhyay *et al* 1997) are not suited for the production of ceramic microcomponents, as they still do not possess sufficient resolution and accuracy or do not provide prototypes with the properties of conventionally shaped ceramics (Heinrich 1999; Varadan *et al* 2001). As far as resolution is concerned, stereolithography-based processes are superior to other techniques and may yield good results in special cases (Griffith & Holloran 1996; Doreau *et al* 2000), but the thermal treatment of the cured ceramic-polymer-composites still seems to be a challenge (Zhang *et al* 1999; Dufaud & Corbel 2001; Provin *et al* 2001). This problem can be bypassed by establishing a rapid prototyping process chain (RPPC) consisting of an RP method as a fast and inexpensive supply for polymer master models and a ceramic shaping method that enables the replication of the RP model into multiple ceramic materials within a short time (Knitter *et al* 1999).

2. Rapid prototyping process chains

The manufacturing of ceramic microparts presented here set out with the 3D-CAD construction of the desired part. The data are transferred to a rapid prototyping equipment, where a model is fabricated out of polymers. The polymer master model is then moulded with liquid silicon rubber, which is subsequently used as a tool in the low-pressure injection moulding (LPIM). The final ceramic part is obtained after dewaxing and sintering. The different steps of the RPPC are shown in figure 1.

2.1 *Fabrication of the master model*

For fabrication of the master models different rapid prototyping methods are applied, where CAD data are used to build a precise master model in a generative process layer by layer. For the accurate dimensionality of the ceramic part to be produced it is important that the CAD model is previously enlarged to a certain degree to take into account the shrinkage of the ceramic part during later thermal treatment. In addition to stereolithography (SLA), we also used multi-jet modelling (MJM) and Rapid Micro Product Development (RMPD) as rapid prototyping processes.

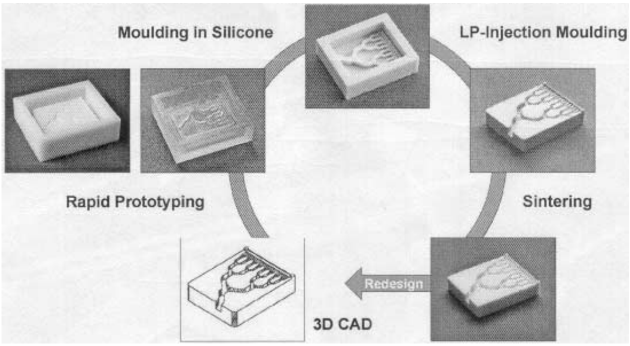

Figure 1. Process steps of the rapid prototyping process chain for the fabrication of ceramic microcomponents.

A commercial stereolithography facility (FS-REALIZER, Fockele & Schwarze, Germany) was used to produce epoxide master models. This facility has a positioning accuracy of 10 μm, and a solid-state laser with a spot diameter of 100 μm is applied to expose a liquid monomer on a construction platform in a scanning mode layer by layer. The exposed spots are subject to polymerisation. Prior to the curing of the next layer, the construction platform is lowered further into the liquid monomer in steps of 50 or 100 μm. In a first approximation, the achievable geometric resolution depends on the layer thickness, the beam diameter, and via beam intensity on the interaction volume of the monomer.

If small-sized holes or narrow trenches are made by stereolithography, problems arise from partly cured resin in narrow gaps. After removing the part from the resin bath, these details cannot be cleaned sufficiently from adherent resin residues and the parts are hence replicated with poor accuracy. For the fabrication of such items a ballistic method like MJM should be preferred. Thermopolymer models were made by this method (ACTUA 2100, 3D Systems) with a drop size of 90 μm and a layer thickness of 40 or 120 μm. However, these models are less suited for microfabrication because of the inherent limited accuracy and a relatively high surface roughness.

For higher resolution, master models were made of acrylates using the RMPD technique at microTEC, Germany [http://www.microtec-d.com]. This modification of stereolithography, which is suited for microdimensioning, allows the reaching of a precision of about 5 μm and enables parallel fabrication of a number of components. Objects with a layer thickness of only about 1 μm may be generated. Besides selective irradiation, parts may also be produced by the RMPD mask technique. This is a combination of mask technology used in photolithography and RMPD, which allows large-area exposure of the monomer layer, and a considerable reduction of exposure times of 2·5-dimensional geometries. The used master models were fabricated with a layer thickness of 25 μm.

In the next step the polymer master models are moulded with liquid silicone rubber which can be easily demoulded after curing. Nevertheless, different silicone rubber materials may be selected to fulfil the specific demands of different designs. The silicone castings are the negative moulds which are subsequently used as moulding tools for low-pressure injection moulding. Contrary to master models made of thermopolymer, whose tiny details are often destroyed when moulded into silicone, less brittle epoxide master moulds are particularly suited to produce a large number of silicone castings.

2.2 *Low-pressure injection moulding*

The prerequisites to use soft silicone moulds as tools in the ceramic shaping process are sufficient low pressure and temperature conditions during moulding. A process that is particularly suited for this purpose is low-pressure injection moulding (Peltsman & Peltsman 1981, 1982; Lenk 1995). Contrary to the better known high-pressure injection moulding, where the feedstock is plasticised by thermopolymers of high viscosity, in LPIM, waxes and paraffins are used as binders. Due to their low melting points moulding can take place at temperatures of about 80–100°C and at pressures below 0·7 MPa (Mangels 1994).

For accurate reproduction of microdimensioned details, ceramic powder with sufficient by small grain size has to be selected. As a rule of thumb, the grain size in the sintered part should be ten times smaller than the tiniest detail to be moulded. However, with decreasing grain size, the specific surface of the particles increases and more organics are needed for proper feedstock preparation. Consequently, the green density is lower and the shrinkage increases. On the other hand, the shrinkage of the parts during thermal treatment should be as low as possible to minimise the standard deviation of the dimensions (Corbett & Schaffer 1987). Furthermore the viscosity of the feedstock has to meet the requirements of the moulding process. The viscosity has to be low enough to fill the small trenches and gaps in the mould and to ensure the release of entrapped gas bubbles from the mould. For alumina feedstock, for instance, 88–91 wt.% of a ceramic powder with a mean particle size of 1·2 μm (MR 52, Martinswerk, Germany) are dispersed in a molten mixture of 8–11 wt.% of paraffin and about 1 wt.% of an emulsifier. The ceramic amounts correspond to green densities of 62–70% of the theoretical density of alumina (Risthaus *et al* 2001). In contrast, solid contents of submicron zirconia (YZ01, SEPR, France, $d_{50} = 0{\cdot}6\,\mu$m) (Bauer *et al* 2001a) and PZT (Megacera D, Megacera Inc., Japan, $d_{50} = 0{\cdot}3\,\mu$m) feedstocks amount to only 50–60 vol.%.

The filling of the silicone mould takes place in a commercial injection moulding facility (Peltsman, Minneapolis, USA or GOCERAM I. V., Moelndal, Sweden) (figure 2). For complete filling of the mould, the tool has be evacuated prior to injection and the mould should to be heated to a temperature that exceeds the melting point of the paraffin. Due to the elasticity of the silicone mould, it is also required to adapt the machine parameters in order to ensure sufficient dimensional accuracy. In case of the fabrication of only a few samples, manual filling of the mould may be reasonable. For this purpose, the pasty feedstock is cast

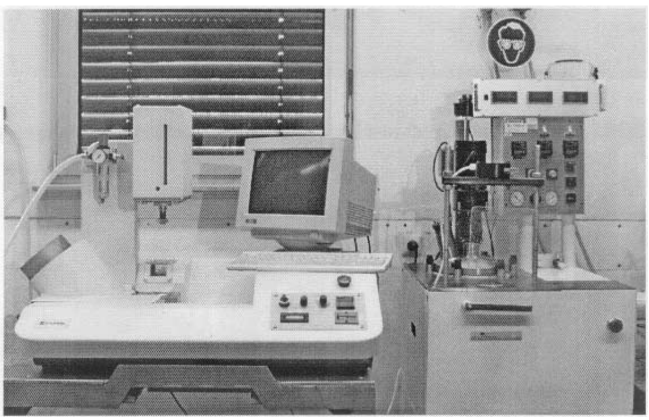

Figure 2. Low pressure injection moulding machines from GOCERAM I. V. (left) and Peltsman Corp. (right).

into the mould at a temperature of about 100°C. Air inclusions are eliminated by repeated evacuation.

According to the experience gained so far, each silicone mould usually allows more than 100 ceramic parts to be produced. Hence, with multiple copies of one master model the silicone moulds may be applied for the production of larger series. As a result of the negligible affinity of silicone rubber to paraffin and of the elasticity of these moulds, demoulding of fragile structural details of ceramic green compacts is simplified considerably. It even becomes possible to demould slight undercuts without a complex tool design. As far as macroscopic parts are concerned, moulding of the ceramics has already been demonstrated using polymer moulds produced by stereolithography directly into silicone without a replication step. In that case a mould release has to be applied prior to each moulding process (Judson & Starr 1999). In case of moulding microcomponents, however, mould release agents may not be employed, because the film thickness can no longer be neglected and would lead to inaccurate reproduction of edges.

After demoulding, organic additives are removed from the green bodies by a slow heating process of up to 500°C. Heating rates and dwell times are strongly dependent on the geometry and design of the components. For large or thick-walled components, the binder burnout should take place with slower heating rates than in case of small or thin-walled parts. Edges or corners should be avoided in the design as at these points increased stresses could occur and may cause cracks during thermal treatment. The parts are sintered in a conventional manner. For example, sintering of alumina takes place at temperatures of up to 1700°C. The total duration of the thermal treatment is in the range of 20 to 30 h. A density of 97% was typically achieved in the sintered alumina components. Linear shrinkage of these parts amounts to about 12%. Owing to the lower green density of the feedstocks, linear shrinkages of the zirconia and PZT components are in the range of 15–20%.

3. Results and discussion

Due to the relatively simple feedstock preparation, the low-pressure injection moulding process may be applied easily and rapidly to different ceramic materials. Components have been produced from Al_2O_3, ZrO_2, $BaTiO_3$, PZT, hydroxyapatite, and an electrically conductive Al_2O_3/TiN ceramic by means of the RPPC (see figure 3). The development of an alumina

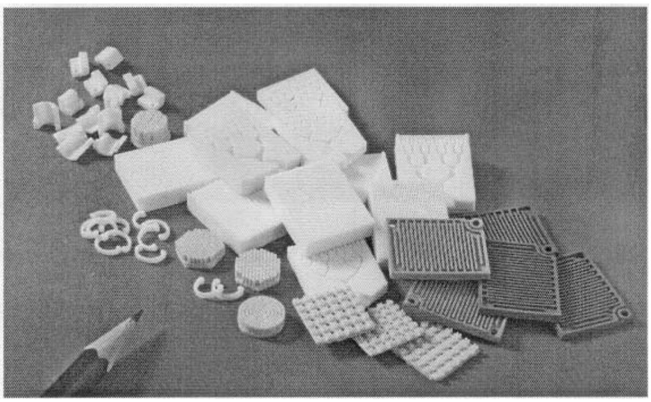

Figure 3. Microcomponents made of different ceramic materials fabricated by the RPPC.

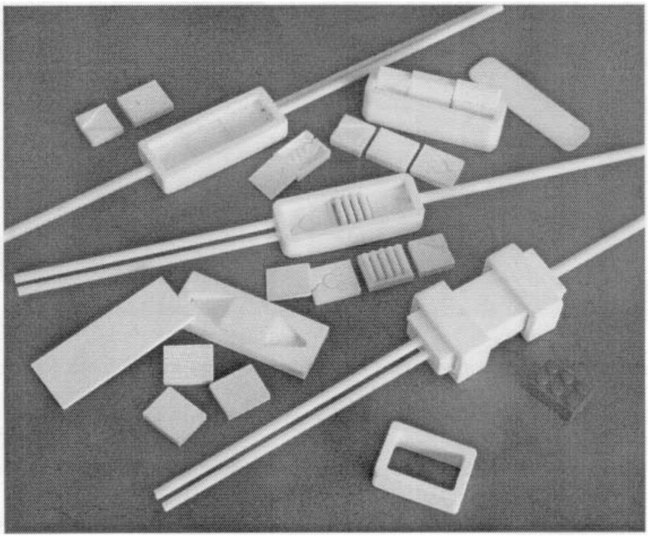

Figure 4. Modular ceramic microreactor systems developed and fabricated using the RPPC. All reactor housings have a length of 68 mm.

microreactor for the investigation of high-temperature gas-phase reactions can demonstrate the distinctive advantages of the RPPC (Knitter *et al* 2001a). Whereas metallic microreactors have already proved to work successfully in microreaction technology, comparable ceramic components for very high temperatures or corrosive conditions are still lacking (Ehrfeld *et al* 2000). Due to contrary material properties as well as different fabrication and joining techniques, at first a novel design had to be established for the ceramic microreactor components. During the design development and due to the desired modular character of the reactor, a variety of moulding tools were needed. The manufacturing of functional models was indispensable, as the final design could not be verified on design models, but only under operating conditions at temperatures of up to 1000°C. Moreover, a fabrication technique had to be chosen that met the requirements for the moulding of the relatively large reactor housings as well as of the micropatterned details of the modular components. A variety of modular microreactor systems is shown in figure 4. All reactor components except the media supply tubes were fabricated by the established process chain, using SLA master models.

As a whole, the replication steps of the rapid prototyping process chain exhibit high precision and accuracy in the micrometer range. Measurements with regard to the reproducibility of the dimensions of structural details yield a standard deviation of only 0·2% for feedstocks of high ceramic loadings. The achievable resolution and surface quality are mainly limited by the layer structure of the RP part, where each inclined area of the component is approximated by steps. In addition, the layer structure of the master model is reflected by periodic depressions on the vertical walls. In a fluidic component with constantly decreasing channels made of alumina, the layer structure of the master model is clearly visible at the vertical walls and as steps at the bottom of the channels (see figure 5). But it should be emphasised that this apparent layer structure of the ceramic component (see insert in figure 5) cannot affect a delamination, as it is only caused by the replication of the surface characteristics of the master model.

The high moulding precision of the RPPC is favourably displayed by the comparison of the three moulding steps. In figures 6 a-c the polymer master model, the silicone mould, and

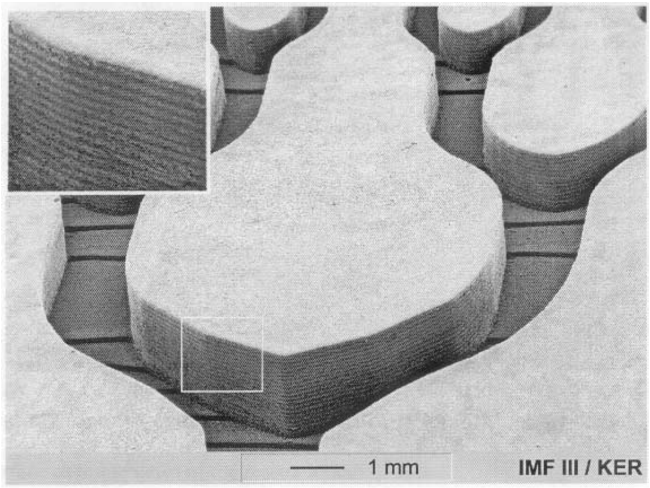

Figure 5. An alumina fluidic component made by replication of an SLA master model. The insert shows the replication of the layer structure of the master model.

the ceramic component are represented based on the faulty example of an alumina fluidic component. For better comparison, the image of the silicone mould has been mirrored. Some small defects in the polymer master model that arose from an inprecise laser control are copied into the silicone mould as well as into the alumina component. In contrast to the component in figure 6c the alumina part in figure 6d is moulded from a defect-free master model. It is evident that the moulding capability is higher than necessary for the moulded details here and that the quality of the RP model is of decisive importance for the quality of the fabricated ceramic component.

The use of the RPPC has also been proved advantageous for the development of piezoelectric components. For a fingerprint scanner PZT, transducers were developed with designs that

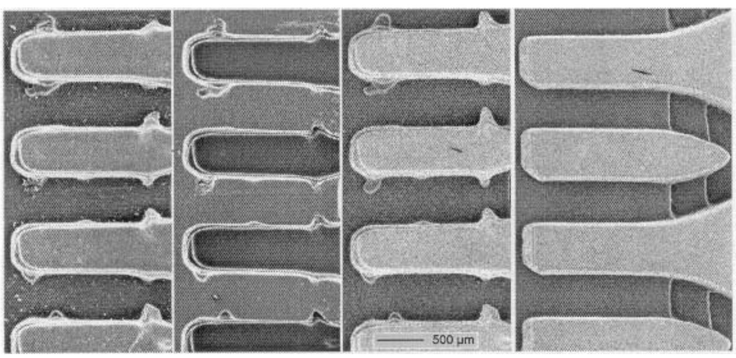

Figure 6. Comparison of the different moulding steps, **(a)** polymer master model, **(b)** mirrored image of the silicone mould, **(c)** sintered alumina part. The defects in the master model are affected by an incorrect laser control in stereolithography and are replicated throughout the process chain into the ceramic part. **(d)** A ceramic part moulded from a defect-free master model.

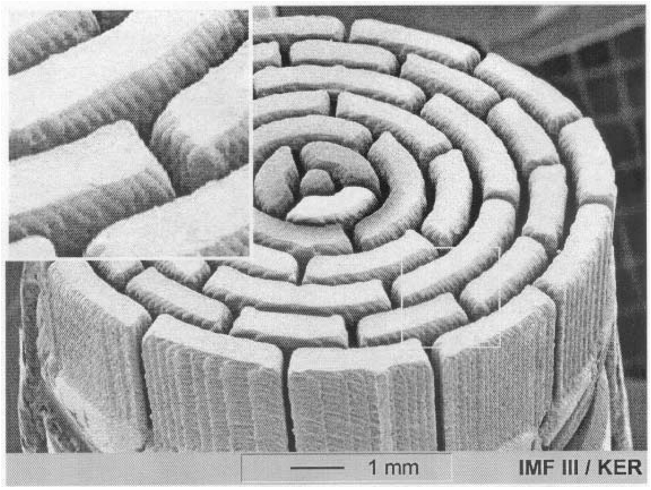

Figure 7. PZT transducer for a fingerprint scanner, replicated by an MJM master model. The insert shows the replication of the surface characteristics of the master model.

could not be made by the usually applied dice and fill method. As the theoretical background for this principle is still lacking, different prototypes were indispensable for the optimisation of the concept. During the preparation of the master models by stereolithography it became evident that it was not possible to remove the remaining, partly cured monomer completely out of the narrow gaps of only 200 μm with an aspect ratio of 10. Hence, in this case MJM was used for the fabrication of the master models in spite of the lower resolution. The PZT part in figure 7 is moulded with high quality, but exhibits a relatively high surface roughness particularly at the vertical walls. For the performance of the piezo components, however, the surface roughness is not crucial. The roughness is here dominated by the relatively low resolution of the MJM process in the x–y plane. Due to the fact that the master model is built by adding drops of molten thermopolymer, the layer structure is less distinct as in SLA master models (see insert in figure 7).

For the fabrication of ceramic components with high aspect ratios the decisive process steps are the filling of the mould and the demoulding. Whereas the filling of the mould can be achieved by an appropriate feedstock viscosity and the evacuation of the mould, the demoulding is the more crucial step. With increasing aspect ratio the friction forces during demoulding will enlarge and can cause damage in both moulding steps, either to the master model or to the silicone mould in the moulding or to the ceramic part in LPIM (Knitter *et al* 2001b). The demoulding is considerably effected by the strength of the demoulded part and the surface quality of the mould, and is noticeably facilitated by the use of elastic materials like silicone. The capability of LPIM to mould high aspect ratios has already been confirmed for moulds with high surface quality (Bauer *et al* 2001b). Due to the fact that the RP models used here are built layer by layer, they exhibit, like all parts made by generative techniques, an inherent surface roughness of the vertical walls. This roughness is influenced by the resolution of the RP technique and the layer height used, but cannot be totally avoided. To evaluate the limits of the RPPC, different test patterns with details down to 50 μm and aspect ratios up to 10 were fabricated with the RMPD technique with a layer height of 25 μm. During the first moulding step into silicone sometimes certain fragile details of the master model were damaged due to the brittleness of the acrylate model used (Knitter *et al* 2001b). This damage could be avoided by the use of a more elastic silicone material. In moulding tests of zirconia

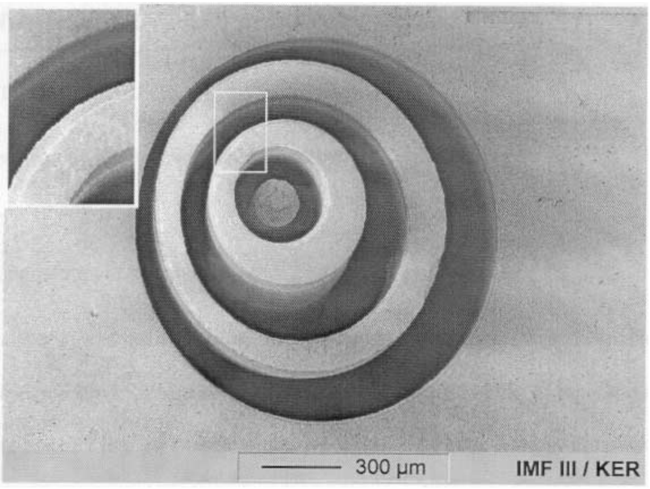

Figure 8. Detailed view of a zirconia test pattern, replicated from an RMPD master model.

and alumina feedstocks, better results could be achieved with the zirconia feedstock due to a higher strength of the green body. Some details of the test pattern moulded in zirconia are shown in figures 8 and 9. The eccentric ring pattern with varying aspect ratios in figure 8 is moulded with a high sharpness of the edges. Even the small misalignment of the last mask during curing of the master model is visible in the moulded ceramic part. The inner column, however, is not moulded defect-free. The upper part of the column is missing, because it was damaged during demoulding. Figure 9 again displays the high moulding capability, as the 25 μm-layers of the master model are clearly visible at the surface of the ceramic component (see insert in figure 9). However, this roughness led to high friction forces during demoulding and aggravated the demoulding of aspect ratios of more than 5. Nevertheless, it was possible to demould aspect ratios of up to 10, though with poor repeatability. The repeatability may

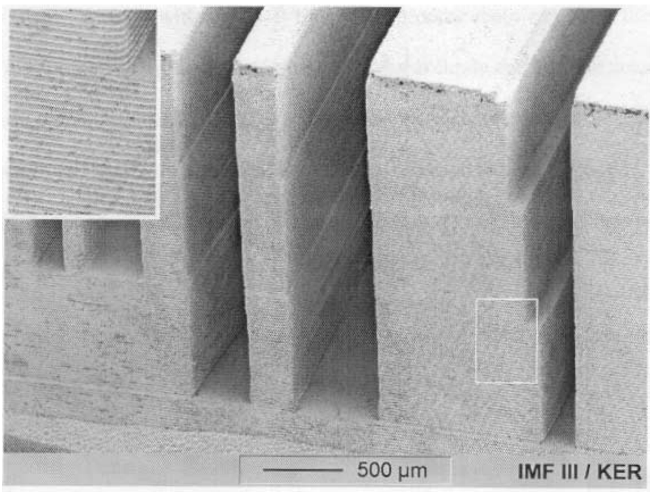

Figure 9. Zirconia test pattern, replicated from an RMPD master model. The insert shows the replication of the 25 μm-layers of the master model.

be improved by a further feedstock optimisation, but aspect ratios of 10 seem to be the limit as long as RP parts are used as master models. However, the aspect ratio is no absolutely valid parameter, as the moulding ability also depends on the geometry. A patterning detail 2000 μm high and 200 μm wide is easier to fabricate than one 500 μm and high 50 μm wide respectively. Furthermore holes and gaps in the ceramic components are easier to mould than walls or free-standing columns. Indeed, up to now such high aspect ratios are quite seldom required for ceramic microparts.

4. Conclusions

During product development, the cost of fabrication of prototypes and functional models is very high. This is especially true for ceramic microcomponents because of the high tooling costs of conventional shaping processes like high-pressure injection moulding. As ceramic parts fabricated by solid freeform fabrication techniques either do not have sufficient resolution or do not provide the final properties of conventionally fabricated parts, they cannot be used in performance tests for design optimisation. However, the advantages of rapid prototyping as an inexpensive and fast supply of master model can be combined in a rapid prototyping process chain with the accurate and flexible low-pressure injection moulding process for ceramic materials. Established RPPC enables a fast supply of ceramic microparts from functional models to pre-production lots and a cost-efficient redesign. Only after design-freeze and for a large number of pieces, where tooling costs play a minor rule, transition to high-pressure injection moulding may be economically feasible. The process chain can be easily applied to various ceramic materials, due to the fast and simple feedstock preparation. As the master models are not directly used as moulding tools but are replicated into silicone moulds, demoulding is simplified and a great variety of differently fabricated master models can be used. Nevertheless, the rapid prototyping technique should be carefully selected to meet the requirements of the ceramic part to be moulded. Because of the excellent moulding capability of LPIM the quality of the master model is of decisive importance for the quality of the ceramic components. Alumina microreactor systems and PZT transducers are examples of various ceramic microparts or micropatterned ceramics, where RPPC was advantageously applied. Without the use of the process chain, these developments would not have been possible within a reasonable period of time and at acceptable costs.

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