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Ceramic micro parts. Part 2: Process-related factors influencing surface finish and shape retention during thermal debinding

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Abstract

Thermal debinding opens up the chance to improve the surface finish of ceramic micro parts. This improvement can be ascribed to the unique effect of surface defect healing. It results in outstanding mechanical properties and was already presented in the first part of this paper. 3Y-TZP micro bending bars were selected as a model and fabricated by a modified option of low-pressure injection moulding (LPIM). The effect of surface defect healing, however, suffers from severe reproducibility of surface finish and shape retention. There are several process-related influencing factors, which have a great impact on the evolution of micro part properties during debinding. With this study, it is intended to give a comprehensive summary about the cause and effects of important process-related factors. Thereby, it is aimed to provide the required background in order to manage the fabrication process and to take advantage of the effect of surface defect healing.

Keywords: Thermal debinding; Zirconia; Low-pressure injection moulding; Micro part; Processing

1. Introduction

Due to the combination of outstanding properties such as high strength and hardness as well as resistance to wear and corrosion, ceramics are gaining more attraction in microsystems technology (MST).[1–4] There are several examples for successful implementation of ceramics in MST, such as fibre optic connectors,[5] wire bonding tools,[6] surgical forceps and scissors,[7] micro reactors,[8] micro pumps,[9] micro motors[10] and micro turbines.[11] As a well-established micro-manufacturing process, micro powder injection moulding (μ-PIM) enables the cost-effective mass production of near-net shaped ceramic micro parts and components for MST[12–14] (PIM is mostly a synonym for high-pressure injection moulding, HPIM). For small-scale production and prototyping, low-pressure injection moulding (LPIM) and hot moulding, respectively, exhibits a promising and low-cost method to realize ceramic micro parts.[15,16] Up to now, a reliable fabrication of ceramic micro parts via LPIM, however, suffered from severe reproducibility of surface finish and shape retention. In this context, thermal debinding was identified as the most important and crucial process step. Usually, tremendous change of surface state and deformation of the fragile micro parts was observed at this stage.[17]

Much effort has been made in order to understand the correlation between processing and the properties of 3Y-TZP micro bending bars selected as a model system.[18–23] A comprehensive understanding about the influence of process parameters on micro part properties, however, was still missing. In the first part of this paper.[24] we have introduced how thermal debinding affects the evolution of the surface state and shape retention of hot moulded 3Y-TZP micro bending bar specimen. With it, the unique effect of surface defect healing and levelling was presented. We showed how thermal debinding can be utilized in order to enhance the surface finish and to improve the mechanical properties of the ceramic micro parts. In the present paper, a comprehensive summary of the state-of-the-art of process-related factors influencing the micro part properties during thermal debinding is presented. Thereby, the required background is provided in order to be able to manage the fabrication process from the feedstock preparation to the thermal treatment and, specifically, to take advantage of the effect of surface defect healing and levelling.

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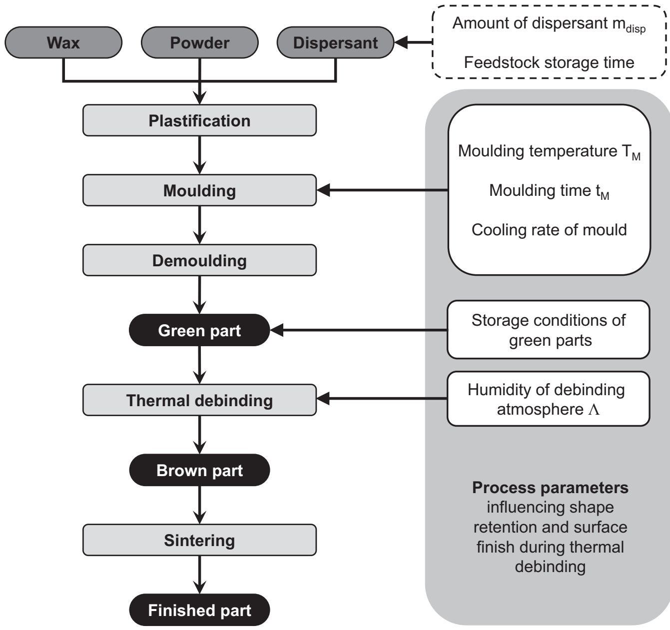

Fig. 1. Process flow: LPIM of zirconia micro parts and relevant process-related influencing factors affecting micro part properties during thermal debinding.

2. Experimental

Zirconia-paraffin wax feedstocks were prepared according to the descriptions given in the first part of this study.[24] The preparation of 3Y-TZP micro bending bar samples by manual hot moulding and the applied characterization methods were also presented. In Fig. 1 the process flow overview of the fabrication method is illustrated. Standard values as well as corresponding variations of applied parameters are listed in Table 1. In a previous work, we already presented several important process parameters.[20] However, the list of relevant influencing factors was not complete at all and a lack of clarity still remained in some points. The ignorance of further important factors made it difficult to allow specific statements about the principle of cause and effect between processing and micro part properties. As a result of this, further experiments were required to find more relevant parameters as well as to obtain a better understanding of the impact of processing on micro part properties. A list of relevant parameters, which, amongst others, were investigated for the first time, is given in Fig. 1 and Table 1. Considering the parameters in Table 1, a controlled fabrication of zirconia micro parts via LPIM can be assured. In order to be able to ascribe changes in micro part property to a specific influencing factor, each process parameter was kept strictly constant, whereas only the relevant parameter was varied, consequently.

Table 1
Standard values and corresponding process parameter variations.

Parameter	Standard value	Variation
Moulding time t_M	3 min	30 min
Moulding temperature T_M	120 °C	80 °C
Cooling condition of mould	Ambient air	Quenching in water (25 °C)
Storage condition of green parts[a]	No storage	Exsiccator (0% RH)/ Ambient air (30% RH)/ Water
Humidity of debinding atmosphere[a]	60% RH	0/10/30% RH

[a] At room temperature (25 °C).

3. Results and discussion

3.1. Wax absorption by moulds

Due to its reusability, cost-efficiency and high moulding accuracy silicone rubber is a predestined mould material for hot moulding of ceramic micro components.[1] However, one major drawback of silicone was found to be the selective absorption of binder components that takes place while it is in contact with the molten feedstock. We reported that paraffin wax is highly absorbed by silicone, whereas it shows no solubility to the dispersant component of the binder.[20] We also found that this effect was one important reason for the poor reproducibility of the shape retention of the micro parts during thermal debinding, especially in terms of edge rounding. Depending on the amount of absorbed wax, it was observed that the edge rounding of the micro bending bars evolves different during debinding. This was attributed to a "liquidation" effect that arises from wax depletion and, as a result of this, enrichment of the dispersant on the surface of the micro parts. This surficial enrichment of dispersant reduces the yield point and causes better flowability of the feedstock in near-surface regions of the micro parts. The effect was found to be controllable by process parameters such as moulding time t_M (representing the time for manual moulding and further evacuation in order to eliminate residual air pockets in the feedstock) and cooling rate of the mould after forming. In general, increasing moulding times t_M and low cooling rates (e.g. at ambient conditions) cause higher tendency to edge rounding (Fig. 2), as the extent of liquidation increases. In the worst case, the liquidation rises to such an extent so that deformation takes place, for example when long moulding times of 30 min are applied (Fig. 2).

Another important process parameter, namely the moulding temperature T_M, was, however, not considered yet. According to this, it is also possible to control the wax diffusion from the molten feedstock into silicone by changing the moulding temperature. It was found that a reduction of T_M from 120 °C to 80 °C results in less pronounced edge rounding (Fig. 2). In this case, the effect of liquidation is minor, as wax diffusion into the mould is limited. However, the extent of deformation or shape retention also depends on the amount of dispersant used for the feedstock formulation. For high amounts of dispersant the extent of edge rounding or deformation is likely to be higher, as the impact of liquidation is expected to be higher for more flowable feedstocks. This correlation is illustrated in Fig. 3a, where the influence of the aforementioned process parameters on the edge rounding is presented in dependency on the amount of dispersant. For increased moulding time t_M (30 min) the tendency to edge rounding and deformation is much higher with increasing amount of dispersant than for the standard value (3 min). In contrast, parameter changes limiting the wax