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Ceramic micro parts. Part 1: How thermal debinding can be utilized to enhance surface finish and mechanical properties

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Abstract

Thermal debinding represents the most critical processing step of powder injection moulding (PIM) of ceramics. Defects such as cracks and pores might be caused, when the process cannot be controlled properly. Considering low-pressure injection moulding (LPIM), however, thermal debinding opens up the possibility to enhance the mechanical properties of ceramic micro parts. In this study, the unique effect of surface defect healing is presented, which takes place during debinding. It enables improved surface finish and results in increased mechanical strength and reliability. As a model, 3Y-TZP micro bending bars with dimensions of 200 μm × 200 μm × 1200 μm were selected. It could be revealed that thermal debinding can be utilized to increase the characteristic 3-point-bending strength up to 3235 MPa with Weibull modulus of 21.4. This result corresponds to macroscopic bending strength of 1727 MPa, which can be achieved only by exhausting fabrication methods and surface post-processing.

Keywords: Thermal debinding; Zirconia; 3Y-TZP; Low-pressure injection moulding; Micro part

1. Introduction

As microsystems technology (MST) is growing progressively, the need for miniaturization of technical devices has increased in the last decade and, with this, the interest in fabrication of micro parts and components.[1] Micro powder injection moulding (μ-PIM) is a well-established micro-manufacturing process for micro parts and components.[2–5] Owing to their particularly good mechanical, thermal, chemical and other special physical properties, the demand especially for near-net-shaped ceramic micro components is increasing rapidly.[6] The most important sectors for ceramic micro parts are medical technology, mechanical and electrical engineering.[7] Examples for high precision micro components for microelectronics are fibre optic connectors[8] and semiconductor wire bonding tools.[9] For medical and dental applications, orthodontic brackets, surgical forceps and scissors for minimally invasive endoscopy can be mentioned.[9,10]

Usually, μ-PIM is applied as a micro-manufacturing process for mass production (PIM is mostly a synonym for high-pressure injection moulding, HPIM). However, there is also need for customized ceramic micro parts as prototypes as well as in small series.[11] In order to satisfy these requirements, low-pressure injection moulding (LPIM) is applied as an alternative micro-manufacturing method. LPIM is sometimes also referred as hot moulding[12] and is much more cost-effective than HPIM.[13] For LPIM, the use of low-melting waxes as major binder component results in feedstocks with low viscosities in the range of 1–20 Pa s (at 100 s^{-1}), which are typically processed at low temperatures of about 90 °C (for HPIM: 100–1000 Pa s at 100 s^{-1} and processing temperatures of 120–200 °C). This is a prerequisite for the application of injection pressures lower than 5 MPa. For HPIM, typically, injection pressures higher than 50 MPa are required. Consequently, low tooling costs, simple and re-usable moulds such as soft silicone moulds predestines LPIM as a micro-manufacturing process for prototypes and small series production.[3] Using rapid prototyping process chain (RPPC) techniques,[6,14,15] it is possible to realize near-net-shaped ceramic parts in the micron range in a very fast and cost-effective way. Recently, this was demonstrated in a successful case study on ceramic micro turbines.[16]

In general, the low viscosities and yield strengths of LPIM feedstocks enable good mouldability and complete mould filling in the micron range at low injection pressures and temperatures. However, this advantage is accompanied by a major drawback considering the mechanical strength of the green parts.

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After mould filling the green parts have to be demoulded and handled carefully in order to avoid any damage of the fragile micro parts and structures. Additionally, the reduced mechanical strength of the green parts often results in deformation of the green micro parts during thermal debinding. LPIM feedstocks melt over a narrow temperature range, where the green parts promptly become soft and deformable. Therefore, the thermal treatment is much more complicated than for green parts fabricated via HPIM, which are usually stabilized by thermoplastic backbone polymers such as polyethylene. Even the successful thermal debinding of typical macroscopic green parts fabricated via LPIM is always considered to be a great challenge.[5,17]

Nevertheless, it is possible to turn this disadvantage and challenge of LPIM into a chance for improving the surface finish and, with it, the strength and reliability of micro parts and components. During thermal debinding, a unique effect can be exploited, which we call surface defect healing and levelling.[18] Taking advantage of this effect is only possible under the precondition that the surface-to-volume ratio of the components is high enough, which is given for micro parts. The enhancement of the surface finish of micro parts by a levelling process is, however, usually associated with rounding of sharp edges and contours. In the worst case, exceeding deformation takes place and it is not possible to keep the geometrical integrity of the micro parts.[19] In order to enable shape retention as well as an improvement of the surface finish and, as a result of this, the mechanical properties of the micro parts, a precise understanding of the micro specific processes during thermal debinding is necessary. In several studies the relationship between the processing via LPIM and the properties of 3Y-TZP micro bending bars, such as surface roughness, edge rounding and strength, were investigated.[18,20–27] Until recently, however, it was not possible to reproduce the properties of the micro parts efficiently, as type and impact of important manufacturing conditions were largely unknown.

In the present paper, which is intended as the first of two parts in this issue, the effect of surface improvement and defect healing during thermal debinding is discussed in detail. As a major material-related factor, the influence of the amount of dispersant on the feedstock flow behaviour and the properties of 3Y-TZP micro bending bars is presented. In the second paper, the state-of-the-art of process-related factors affecting the micro part properties is presented. Therein, it is intended to provide the required background to be able to control the fabrication process and to take advantage of the effect of surface defect healing in order to enhance the mechanical properties of low-pressure injection moulded ceramic micro parts efficiently.

2. Experimental

2.1. Sample preparation

Zirconia powder (TZ-3YS-E, Tosoh, Japan), paraffin wax (TerHell 6403, Schümann Sasol, Germany) and a commercial dispersant (Hypermer LP1, Croda, UK) were used as starting materials for preparing zirconia–paraffin feedstocks. The solids loading was kept constant at 50 vol% (corresponding to consolidated state at room temperature), whereas the amount of dispersant varied from 1.6 mg/m^2 to 3.2 mg/m^2 (mg surfactant per m^2 surface of powder), as already described in previous works.[19,28] The powder was dried at 300 °C for 2 h prior to homogenization and was kept at 120 °C before adding to the molten binder. A laboratory vacuum dissolver (Dispermat, VMA-Getzmann, Germany) was used for plastification and homogenization of the powder–binder mixture at 100 °C. Micro bending bars were fabricated according to the rapid prototyping process chain (RPPC) for ceramic micro components proposed by Bauer et al.[6] and Knitter et al.[14,15] A micro-milled brass array consisting of 15 × 15 columns with square cross sections of about 250 μm × 250 μm × 1500 μm was applied as a master model for the casting of soft silicone rubber moulds (Elastosil M4600 A/B, Wacker Chemie, Germany), which were used, in turn, for the replication procedure by LPIM. For the brass master model, a silicone mould and a micro bending bar array in green as well as in sintered state it is referred to our previous study.[19] Applying a manual option of LPIM (sometimes also referred as manual hot moulding), the feedstocks were moulded manually into the preheated moulds by using a spatula and, immediately, were evacuated under vibration to achieve an adequate filling of the moulds. This pressureless moulding technique was possible due to the low viscosities of the feedstocks.[28] It was favoured over a semi-automatic LPIM machine, as the required feedstock amount is significantly smaller than using a LPIM machine and, thus, more suitable in laboratory scale. After moulding and evacuation, the moulds were cooled down to room temperature at ambient air. The manual moulding procedure was conducted at 120 °C (total solid content at this temperature was calculated as 46.1 vol%) and the total time for moulding, evacuation and vibration procedure was set as 3 min. Afterwards, the green parts were manually demoulded and the bulk support was smoothened by grinding. In Fig. 1 the process chain steps are summarized and illustrated schematically. The used silicone moulds were cleaned with compressed air and treated with a modelling wax (Freeman 6549-D NYC-Pink, Freeman Manufacturing and Supply Company, USA) in order to clean residues of the demoulded green parts in the micro cavities. Thermal debinding was performed on porous alumina plates (Keralpor 99, Kerafol, Germany) with 0.5 K/min up to 500 °C and a dwell time of 10 min by using a debinding furnace with adjustable circulation and humidity of the air in the furnace (HT6/28, Carbolite, UK). Sintering in air was performed with 3 K/min up to 1450 °C and a dwell time of 1 h (VMK1800, Linn High Term, Germany). After sintering, the micro bending bar columns exhibited dimensions of approximately 200 μm × 200 μm × 1200 μm.

2.2. Characterization methods

The rheological properties of the zirconia–paraffin feedstock formulations were analysed by using a rotational rheometer and a plate–plate measuring system with plate-to-plate distance of 0.5 mm. The averaged roughness height R_Z (DIN 4768)[29] of green and sintered micro bending bars was determined by using a non-destructive metrological surface measuring system with a chromatic white light sensor (MicroProf, Fries Research &

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