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Binder removal via a two-stage debinding process for ceramic injection molding parts

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Abstract

Debinding binders in two stages is critical to maintaining the shape of injected parts; the resulting decomposition affects the strength and rigidity of a structure. This study determines the optimal debinding process on the basis of a higher binder removal rate and the production of defect-free parts. The feedstock used was a combination of alumina–zirconia powder with a binder that consists of high-density polyethylene (HDPE), paraffin wax (PW), and stearic acid (SA). During the first stage, the injected parts were immersed in an *n*-heptane solution at 50 °C, 60 °C, 65 °C, and 70 °C to remove PW and SA. Binder weight loss was evaluated as a function of time. In the second stage, HDPE was removed by using thermal debinding. The results show that the optimum solvent debinding process runs for 16 h at 60 °C. The weight loss of the binder reaches 41.1% and results in the formation of defect-free parts. The binders are degraded at approximately 550 °C during thermal debinding. This degradation resulted in decomposition of nearly 96.9% of the binders. Low heating rates (1 °C/min to 2 °C/min) prevent defects from forming in the injected parts.

Keywords: A. Injection molding; C. Diffusion; Debinding; Alumina–zirconia

1. Introduction

Ceramic injection molding (CIM) is a combination of powder technology and injection molding. The CIM process is a near-net shape processing technique that facilitates the low-cost manufacture of ceramic components with complex shapes [1–3]. The CIM process involves several stages, namely, mixing, injection molding, debinding, and sintering. German and Bose [4] described debinding as a process in which a binder is removed from injected parts, thereby producing commonly designated brown parts. The process must be carefully performed to avoid problems that affect quality, such as component distortion, cracking, blistering, and contamination of parts. Debinding involves a long processing period, thereby prompting the development of different debinding techniques, including solvent, thermal, wicking, evaporation and catalytic debinding, as well as the combination of these approaches [5,6].

The use of a multi-component binder system enables two-stage binder removal. German [7] and Liu et al. [8] stated that binder removal in two stages is important in avoiding defects in the shape of parts and in reducing total debinding time. The main binder (backbone component) is usually a thermoplastic that maintains the shape of the injected parts by confining ceramic powder particles, which are then thermally removed during the second debinding stage. Moreover, the support binder (commonly a wax), which functions as a filler phase, is eliminated during the first debinding stage by immersing the injected part in a solvent, such as heptane, hexane, and kerosene. The low decomposition temperature and molecular weight of the support binder facilitate debinding at the first stage, as reported by Iriany [9] and Krauss et al. [10]. Furthermore, pore channels that are formed as a result of filler removal enable the main binder to seep out of the body structure [11,12].

Cheng et al. [13] and Thomas-Vielma et al. [14] showed that combining solvent and thermal debinding techniques successfully

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reduces binder decomposition time and prevents defects from forming in injected parts. However, few researchers have discussed a debinding process that involves the combination of alumina and zirconia ceramic powder with a binder that consists of high-density polyethylene (HDPE), paraffin wax (PW), and stearic acid (SA). In the current study, an *n*-heptane solution was used to dissolve PW and SA during the first debinding stage, and HDPE was removed by thermal debinding during the second stage. The debinding profile for thermal elimination was optimized through thermogravimetric analysis (TGA) of the binder. This study attempted to determine the optimal debinding process based on higher binder removal rate and production of defect-free parts in a multi-component binder system.

2. Materials and methods

2.1. Materials

The feedstock used in this study was a mixture of alumina and zirconia (3 mol% yttria) powders combined with a multi-component binder system composed of HDPE, PW, and SA. The mixture was 80 wt% alumina powder and 20 wt% zirconia powder. The alumina powder (AL-160SG-1), which has an average particle size of 0.40 μm and a specific surface area of 7.0 m^2/g, was supplied by Showa Denko. The zirconia powder (KZ-3YF), which has an average particle size of 0.35 μm and a specific surface area of 9.0 m^2/g, was supplied by KCM Corporation. Before mixing, the ceramic powder was dried for 1 h in an electric furnace at 110 °C. The alumina and zirconia powders were then mixed by using dry mixing, which was performed at 100 rpm for 8 h by using a ball mill. The ball-to-powder ratio was 5:1. The average size and density of the alumina–zirconia powder after dry mixing were 0.31 μm and 4.46 g/cm^3, respectively. The morphology of the alumina–zirconia powder is shown in Fig. 1. Table 1 shows the characterization results for the binders. Differential scanning calorimetry (DSC) analysis and TGA were conducted to determine the melting and decomposition temperatures of the binders. DSC and TGA were performed on a Mettler Toledo DSC 1 STAR^e System and Netzsch STA 449 F3 Jupiter at a heating rate of 10 °C/min.

2.2. Feedstock preparation and injection molding

The alumina–zirconia powder was mixed with the binders by using an internal mixer machine (Brabender W 50 EHT) to produce the feedstock. Mixing was conducted at 140 °C with 20 rpm velocity for 30 min. The alumina–zirconia powder loading was 57 vol% (86.5 wt%), and the binder composition was 50 wt% HDPE, 46 wt% PW, and 4 wt% SA. The composition and combination of the binders were based on the method proposed by Thomas-Vielma et al. [14]. The feedstock, which was appropriately granulated after mixing (Strong Crusher TSC-5JP), consisted of 18.09 wt% carbon, 30.63 wt% oxygen, 36.74 wt% alumina, and 14.54 wt% zirconia. The elemental content, which refers to the average percent of elements with different batches of mixing, was determined by using energy dispersive X-ray spectroscopy (EDX) associated with field emission scanning electron microscopy (SEM) (Hitachi SU8020 FESEM). An SEM image of the feedstock is shown in Fig. 2. A standard screw-type injection molding machine (Battenfeld BA 250 CDC) was used to produce the injected parts. The mold cavity was characterized by a round bar (∅15 × 21 mm^2). The injection temperature and pressure were 160 °C and 110 MPa, respectively. The injection and holding times were 5 s and 10 s, respectively, and the molding temperature was 50 °C. The injection molding conditions were optimized according to previous research [15].

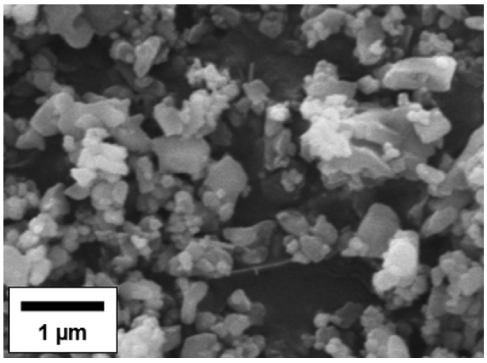

Fig. 1. Morphology of the alumina–zirconia powder.

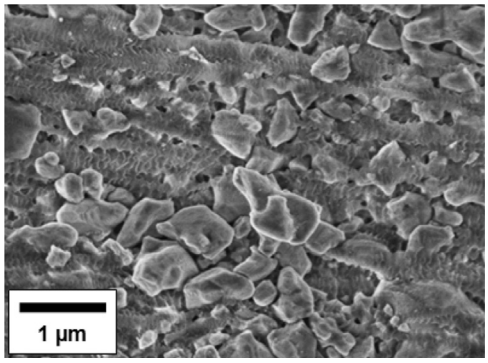

Fig. 2. SEM image of the feedstock.

Table 1
Characterization of the binders.

Binder	Supplier	Chemical structure	Density (g/cm^3)	Melting temperature (°C)	Decomposition temperature (°C)
HDPE	Titan petchem	$(–CH_2–CH_2–)_n$	0.96	131.8	420–550
PW	Emercy oleochemicals	$C_{31}H_{64}$	0.89	59.5	200–400
SA	Emercy oleochemicals	$C_{18}H_{36}O_2$	0.88	69.8	180–380