



Fabrication of zirconia-toughened alumina parts by powder injection molding process: Optimized processing parameters

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Received 21 April 2013; received in revised form 4 May 2013; accepted 22 May 2013
Available online 13 June 2013

Abstract

Zirconia-toughened alumina (ZTA) parts were fabricated by using a powder injection molding process that utilizes a multi-component binder system based on high-density polyethylene, paraffin wax, and stearic acid. The entire aspect of the manufacturing process, which includes mixing, injection molding, debinding, and sintering, were optimized in this study. ZTA powder was mixed with binders at powder loading ranging from 53 vol% to 59 vol%. The optimum powder loading was determined by analyzing the rheological properties and homogeneity of feedstocks. During injection molding, the temperature and injection pressure parameters were manipulated to obtain optimum density results. A two-stage debinding process (solvent and thermal) was used to remove binders in green parts. Debound parts were sintered at temperatures ranging from 1400 °C to 1600 °C for 2 h. The shrinkage, density, and hardness of the sintered parts were measured. Results show that with homogeneous mixing, the feedstocks were transformed into pseudoplastic in less than 30 min. Powder loading of 57 vol% is the most optimal case for injection molding according to the power law index and flow activation energy values. The theoretical relative density reached 90.27% with defect-free parts under optimum injection temperature and pressure. The weights of the parts decreased by 82.26% during solvent debinding at 60 °C, whereas the binders were completely degraded at approximately 550 °C during thermal debinding. Experimental results also indicate that the shrinkage, density, and hardness reached their maximum values at a sintering temperature of 1600 °C. The sintered parts were densified with approximately 98% theoretical density, hardness of 1582.4 HV, and 15% shrinkage value.

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Keywords: A. Injection molding; A. Mixing; C. Mechanical properties; Zirconia-toughened alumina

1. Introduction

Powder injection molding (PIM) is a combination of powder technology and injection molding that involves several stages, including mixing, debinding, and sintering [1]. During mixing, ceramic powder is blended with binders to form a homogeneous compound. Binders provide viscosity to the powder, thereby simplifying the process of filling feedstock into molds during injection molding. In addition, binders help maintain the original shape of the ceramic powder during debinding and until the start of the sintering process [2]. Optimum powder

loading ratio is also important in the success of PIM. The powder and binder ratio ranges from 45% to 75% by volume [3]. A high powder loading ratio will cause inconsistencies in the injected parts, which can subsequently damage the injection machine. By contrast, a low powder loading ratio can cause separation of binders from powder during injection, thus prolonging debinding and leading to considerable shrinkage during sintering [4,5]. An optimum percentage of powder loading can minimize shrinkage, prevent cracking, and increase the mechanical properties of materials [6]. Therefore, the rheological properties of feedstock were evaluated to obtain the optimum powder loading ratio for injection.

The combination of solvent and thermal debinding technique has been proven successful in reducing binder decomposition time and in preventing defects in green parts [7–9]. The use of a multi-component binder system allows binder

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removal in stages (solvent and thermal). Binder removal is important in maintaining the shape of parts, thereby preventing the occurrence of cracking, blistering, and bloating, which may affect the quality of products after sintering [7]. Currently, a method based on powder pressing and slip casting is commonly used in producing zirconia-toughened alumina (ZTA) parts, whereas PIM is still not extensively used. Previous studies have focused only on the use of ceramic powder based on alumina or zirconia materials [6,7,9]. Therefore, this study focused on ZTA ceramic powder because the material properties of ZTA are better than its original nature. A complete PIM process was developed to fabricate cylindrical ZTA parts with the multi-component binder system on the basis of high density polyethylene (HDPE), paraffin wax (PW), and stearic acid (SA). The homogeneity and rheological properties of feedstocks were studied, and the processing parameters for molding, debinding, and sintering were optimized.

2. Material and methods

A combination of alumina and zirconia powders with binders consisting of HDPE, PW, and SA was used as feedstock. The alumina and zirconia powders were mixed at a rate of 80% and 20% by weight, respectively. Alumina powder (AL-160SG-1) with an average particle size of 0.40 μm was supplied by Showa Denko, whereas zirconia powder (KZ-3YF) with an average particle size of 0.35 μm was supplied by KCM Corporation. Table 1 shows the characterization of the binders. Differential scanning calorimetry (DSC) analysis and thermogravimetric analysis (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^c System and Netzsch STA 449 F3 Jupiter at a heating rate of 10 $^{\circ}\text{C}/\text{min}$.

Prior to mixing, ceramic powder was dried in an electric furnace (Medcenter Venticell III) at 110 $^{\circ}\text{C}$ for 1 h. The alumina and zirconia powders were mixed by using the dry mixing method (Fritsch Pulverisette 6), 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 ZTA powder after mixing was 0.31 μm and 4.46 g/cm^3 , respectively. ZTA powder was mixed with binders by using an internal mixer machine (Brabender W 50 EHT) to produce feedstock. Table 2 shows the powder loading ratio and binder composition used in this study. The composition and combination of binders were based on the study of Thomas-Vielma et al. [7]. Powder loading was set based on the critical powder loading (60.5% volume) obtained through the oil absorption method [10]. The feedstocks were labeled AZ53, AZ55, AZ57, and AZ59 according to their percentage of powder loading. German and Bose stated that the optimum powder loading rate is about 2 vol% to 5 vol% lower than the critical powder loading is [11]. The mixing process was conducted at 140 $^{\circ}\text{C}$ with 20 rpm velocity for 30 min. The feedstock was properly granulated with a crusher after mixing (Strong Crusher TSC-5JP).

Homogeneous mixing of feedstock was determined based on torque information recorded in the internal mixer machine [12]. A lower torque value at steady state indicates homogeneous mixing. The homogeneous mixing of feedstocks can also be determined by measuring the standard deviation of density [13]. The density values for each group were obtained from five different feedstock sections by using a Quantachrome Ultrapycnometer 1000 pycnometer. A small standard deviation indicates homogeneous mixing. Feedstock rheological properties were characterized by using a capillary rheometer machine (Shimadzu CFT-500D). A die with 1 mm diameter and 10 mm length was used ($L/D=10$). Tests on rheological properties of feedstock were performed at temperatures ranging from 150 $^{\circ}\text{C}$ to 170 $^{\circ}\text{C}$ with an applied load of 20–90 kgf. Homogeneous feedstocks with good rheological properties were used for injection molding. Energy-dispersive X-ray spectroscopy (EDX) analysis of feedstock for optimal powder loading was evaluated by using a Zeiss Evo MA10 VPSEM.

A standard screw-type injection molding machine (Battenfeld BA 250 CDC) was used to produce the green parts. Mold cavity

Table 1
Characterization of binders.

Binder	Supplier	Density (g/cm^3)	Melting temperature ($^{\circ}\text{C}$)	Decomposition temperature ($^{\circ}\text{C}$)
HDPE	Titan Petchem	0.96	131.78	420–550
PW	Emercy Oleochemicals	0.89	59.53	200–400
SA	Emercy Oleochemicals	0.88	69.83	180–380

Table 2
Composition of feedstocks.

Feedstock tag	ZTA powder/binder (vol%)	Binder (wt%)		
		HDPE	PW	SA
AZ53	53/47	50	46	4
AZ55	55/45			
AZ57	57/43			
AZ59	59/41			

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