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Surface adhesion between ceramic injection molding feedstocks and processing tools

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Abstract

Interfacial adhesion between highly filled aluminum and zirconium oxides ceramic compounds and CIM (Ceramic Injection Molding) processing tools was investigated from contact angle measurement. Polymers considered as binder components were low density polyethylene, paraffin wax, polyethylene glycol, carnauba wax, acrawax, and stearic acid. Channel walls of the mold were constructed from hardened, TiN hardened, nitridized and heat-treated steels. From the calculated surface energies, the superiority of heat-treated steel as well as acrawax and especially polyethylene glycol as binders is derived. Carnauba wax shows similar wettability as stearic acid, thus becoming promising substitute for the role of processing aid. Concerning tested ceramic powders, Al_2O_3 revealed somewhat higher polar component of the surface energy than ZrO_2. The differences in total surface energies of powders and binders are all about 3 J/m^2 lower (PEG) or higher (PW, LDPE, AW, CW, SA) for Al_2O_3 powder than for ZrO_2.

Keywords: Ceramic injection molding; Adhesion; Contact angle; Surface energy

1. Introduction

Ceramic injection molding (CIM) serves as a highly effective technique for manufacturing complex shape parts with high dimension accuracy. This technique also gains superior attention for its ability to reduce production waste and cost by allowing manufacturing parts close to their theoretical net weight. CIM process includes four major stages: (1) mixing; (2) injection molding; (3) debinding; (4) sintering.

The initial stages such as mixing and molding are considered to be of the critical importance, complicated by the necessity of using large number of different materials often with contradicting properties in order to fulfill the processing requirements. Currently, the most efficiency limiting factor of CIM is the separation of powder and binder components during injection molding stage. Thornagel [1] demonstrated that local shear rate gradients are the driving forces initiating phase separation. Assuming no slip condition, i.e. good adhesion of the feedstock to the wall of the channel, a significant shear rate peak occurs close to the wall, while a plateau at much lower shear rate level is observed in the middle of the flow domain. Then, particles flowing in the peak area experience a non-uniform shear rate resulting in their rotation, which becomes severe as the shear stress gradients increase. Such rotating particles try to leave areas of high shear gradients. As a consequence, high shear rate area is characterized by high binder content, while the plateau region of the lower shear rate accommodates a powder rich material.

Recently, we have proposed a method to quantify the separation via SEM/EDX combined with the analytical tool [2] as well as the rheological model suitable for ceramic feedstocks [3]. Further, we have pointed out that wall slip could be considered as a rheological parameter indicating

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powder-binder separation absence/occurrence [4,5]. Nevertheless, the conditions, at which the highly filled materials slip at the wall resulting in a plug flow (and thus no separation), are not only feedstock characteristics and processing parameters, but the most important is an interfacial adhesion between the channel walls and CIM feedstock (binder and powder), depending on the surface treatment of the mold channels.

Therefore, the aim of the paper is to evaluate the adhesion properties of CIM powders and binder components as well as materials most often employed in construction of channel walls of CIM processing tools. In the recent paper [6] the possibility to substitute the polyolefin backbone in alumina feedstocks with polar waxes has been investigated with the regard to debinding and early stage of sintering process. Over 20 feedstocks differing in the aluminum oxide powder loading as well as binder composition containing low density polyethylene or carnauba wax, paraffin wax, polyethylene glycols varying in their molecular weight, and stearic acid as a plasticizer were studied. Adhesion of these components is now considered for four most often used materials for construction of channels and mold-die walls. Contact angle measurement is provided as it has proved itself a method widely employed as a characterization tool in ceramic processing (e.g. [7–9]).

2. Experimental

2.1. Materials

Commonly used CIM binder components – Low Density Polyethylene (LDPE, Laqtene 1200 MN 8, Atochem), Paraffin wax (PW, Paraffinum Solidum, Tamda), Polyethylene glycol (PEG6000, Sinopol, Sino-Japan Chemical), Stearic acid (SA, 95%, Sigma Aldrich), Carnauba Wax (CW, 2442, Kahl), and Acrawax (AW, ACRAWAX C Atomized, Lonza) were studied. Tested materials properties are shown in Table 1. Ceramic powders employed in the study were highly compressive superground aluminum oxide and zirconium oxide powders.

Pure binder components were examined from the flat 1 mm thick samples prepared by a unidirectional pressing at temperatures 20 °C above particular binders' melting points. The contact angles of ceramic powders were studied from the sintered samples prepared as follows: feedstocks (55 vol%) were mixed in a twin screw extruder Brabender (KETSE 20/40) at mixing rate of 150 rpm at temperature of 150 °C, and then molded into a rectangular shapes in an injection molding machine (Allrounder 370S, Arburg). The optimized injection molding parameters are summarized in Table 2. After molding the polymer binder was removed by combined solvent and thermal debinding, and brown parts were sintered to the final densities at maximum sintering temperatures of 1500 °C (ZrO_2) and 1600 °C (Al_2O_3) at a heating ramp of 50 °C/h with 2 h temperature holding followed by cooling down spontaneously.

Four most often used treatments of steel were considered for the discs samples (20 mm in diameter, 2 mm thick) of channel walls of an injection mold: hardened, hardened TiN, nitridized and heat-treated.

Table 1
Properties of tested binder components.

Binder	Abbreviation	Density ISO 1133 [g/cm³]	Melting temperature [°C]	Molecular weight [g/mol]
Low Density Polyethylene	LDPE	0.918	108	250000
Polyethylene glycol 6000	PEG6000	1.21	62	6000
Paraffin wax	PW	0.9	58	400
Carnauba wax	CW	0.97	86	1000
Acrawax	AW	0.99	145	560
Stearic acid	SA	0.85	70	284

2.2. Surface properties determination

Surface properties of the samples prepared from ceramic powders, as well as samples of channel wall materials, were examined with contactless 3D Chromatic Length Aberration (CLA) scanner (Talysurf 300, Taylor and Hobson, UK) equipped with Talymap ver.5.0.2 software. Tested surfaces were subjected to a height measurement over a rectangular area (500 × 500) μm. The data supplied is of the form $z=f(x,y)$, where z is the height of the profile, x stands for the position over the scanning direction, and y corresponds to the number of traces. First Interface Detection (FID) was selected as a measurement mode. The software takes into account the height of the first interference (i.e. the upper border of the transparent interference represented by the first peak in the spectrum).

2.3. Contact angle measurements

The surface energy was determined by measurements of contact angles of three test liquids set (deionized water, ethylene glycol and diiodomethane) using the SEE (Surface Energy Evaluation) system (Advex Instruments, Czech Republic) with the contact angle measurement error $\pm 2°$. A drop of the test liquid ($V=3$ μL) was placed with a micropipette on the material surface, the sessile drop was imaged by a color camera, and the contact angle of the test liquid was measured. For a sessile drop of tested liquid, this is defined as the angle

Table 2
Injection molding parameters.

Parameter	Value Al_2O_3/ZrO_2
Zone 1 temperature (°C)	130
Zone 2 temperature (°C)	135
Zone 3 temperature (°C)	140
Zone 4 temperature (°C)	150
Nozzle temperature (°C)	145
Mold temperature (°C)	30
Injection speed (mm/s)	15
Injection pressure (bar)	1800
Hold pressure (bar)	1500/500
Hold pressure time (s)	2/0.5