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Simulations and injection molding experiments for aluminum nitride feedstock

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Abstract

Powder injection molding (PIM) simulations are useful for predicting mold-filling behavior because they aid in material, process and part design. To perform PIM simulations, measurements for feedstock properties such as physical, thermal, and rheological are required as input parameters. The availability of data for such feedstock properties is limited and fresh measurements are often required in order to perform PIM simulations for variations in feedstock composition. A recent study by our group presented a procedure to estimate feedstock properties and use them in mold-filling simulations. The present work compares the predictions of PIM mold-filling simulations using experimental and estimated feedstock properties with injection-molding experiments. Aluminum nitride (AlN) feedstock of 80.5 wt% was compounded using a twin-screw extruder and injection-molded as tensile bars. Injection-molding experiments were performed using the AlN feedstock at various melt temperatures and injection pressures to obtain complete and partially filled parts. Simulations were performed using measured and estimated AlN feedstock properties on the tensile-bar geometry used during injection molding experiments. Melt temperature was varied while performing simulations to obtain a process window for complete and partially filled parts. A comparison between injection molding experiments and simulations was made to understand the dependence of the estimated and experimental feedstock properties in predicting mold filling behavior.

Keywords: Injection molding; Feedstock properties; Simulation; Aluminum nitride

1. Introduction

Powder injection molding (PIM) is a multi-step process that can be divided into four basic steps: (1) ceramic or metal powders are mixed with polymer binders to form a homogenous feedstock using a twin-screw extruder, (2) the feedstock is injection molded into desired geometries using an injection molding machine, (3) the binder phase is debound from the molded parts by thermal or solvent based techniques, and (4) debound parts are sintered to achieve final parts. The PIM simulations are generally performed typically after feedstock compounding to identify appropriate process and geometry attributes for optimum molding [1–12]. Common injection molding simulation tools include Autodesk Moldflow, Sigmasoft, PIMsolver, and Modlex3D.

To perform PIM simulations, feedstock property measurements for density, thermal conductivity, and specific heat are required for a range of temperatures. Further, viscosity measurements are required for a range of shear rates and temperatures, and specific volume measurements are required for a range of temperatures and pressures [1–4]. Availability of such feedstock property data is limited and properties are typically measured for a particular composition.

A recent work by our group suggests a design method that uses empirical equations and estimate feedstock properties as a function of composition, filler properties, and binder properties [17]. This design method employs the use of available literature filler and binder properties to estimate physical, thermal and rheological properties for nine different ceramic feedstocks [17].

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An extension of this work outlines a procedure to use feedstock property estimates in mold-filling simulations [18].

In the current work, 80.5-weight percent aluminum nitride (AlN) feedstock was extruded and injection molded into a tensile bar geometry. Injection molding was performed as a function of melt temperature and injection pressure in order to obtain parts with partial and complete mold filling. Feedstock properties for AlN were measured at Datapoint Labs and estimated using our design procedure for PIM simulations [5,6]. To perform PIM simulations a tensile bar geometry was designed according to the mold tool dimensions. Simulations were performed using measured and estimated AlN feedstock properties at various melt temperatures and injection pressures to obtain complete and partially filled parts. Comparisons of melt temperatures from simulation and experiments were made to understand the effectiveness of PIM simulations in predicting mold-filling behavior.

2. Experimental methods

Commercially available aluminum nitride (AlN) powder (median particle size of 1 μm) and a wax-polymer binder were used as starting materials. The binder is composed of paraffin wax, low-density polyethylene, polypropylene, and stearic acid. Details of the binder composition and feedstock compounding are provided [19,20]. A feedstock with 80.5 wt% AlN powder was compounded using a 27 mm twin-screw extruder with a length to diameter (*L/D*) ratio of 40.

Material property measurements were made at Datapoint Labs (Ithaca, NY) for the AlN feedstock and a wax-polymer binder [7,8]. Solid density measurements were made for AlN feedstock and wax-polymer binder using Archimedes principle as laid out in ASTM standard D792. The melt density measurement was done using a Gottfert Rheograph capillary rheometer in accordance with ASTM D3835 for AlN feedstock and a wax-polymer binder. A Perkin Elmer differential scanning calorimetry (DSC) was used to measure specific heats for AlN feedstock and a wax-polymer binder in accordance with ASTM E1269 standard. Thermal conductivity measurements for AlN feedstock and the wax-polymer binder were made using a K-System II thermal conductivity system in accordance with ASTM standard D5930. Viscosity for AlN feedstock and the wax-polymer binder was measured according to ASTM D3835 using a Gottfert Rheograph capillary rheometer. Pressure–volume–temperature (PVT) measurements for AlN feedstock and the wax-polymer binder were made with a Gnomix PVT apparatus in accordance with ASTM D792 [7,8]. The feedstock property measurements were compared to values estimated using models listed in Table 1. Description of the symbols used in Table 1 and throughout this paper is presented in Table 2.

The AlN feedstock was injection molded into a tensile bar geometry using an Arburg 221 M injection-molding machine. An injection gate with a size of 6.5 mm was used to inject the AlN feedstock into the mold cavity. Injection molding experiments were performed at a set of melt temperatures and injection pressures to obtain parts with complete and incomplete mold fill (Table 3). In the first molding experiment (Table 3) the injection pressure (P_i) was set at 38 MPa and melt temperature (T_m) was set at 455 K. A total of 50 parts with no defects were molded during this experiment and a specimen part is shown in Fig. 1a. To understand the effect of decreases in injection pressure and melt temperature on the molding behavior, experiments 2–4 were performed (Table 3). For experiments 2–4 the melt temperatures was decreased from 444 K to 422 K while keeping injection pressure constant at 14 MPa to obtain parts with incomplete mold fill (short shot). A total of four to five specimens were injection molded in each of the experiments 2–4 and sample molded specimens are presented in Fig. 1b–d. All injection molding experiments listed in Table 3 were performed at injection speed of 33 cm^3/s, the initial packing pressure was set at 100% of the injection pressure value for the first second and then it was reduced to 65% of the injection pressure value for the next 1.2 s. To calculate the percentage of part filled of the incompletely filled samples, their weights were compared to those of the filled samples.

Table 1
Models used in present study to estimate the feedstock properties.

Property	Empirical relations	
Density	$\frac{1}{\rho_c} = \frac{X_b}{\rho_{b\,exp}} + \frac{X_p}{\rho_p}$	(1)
Volume fraction	$\phi_p = \frac{X_p/\rho_p}{X_p/\rho_p + X_b/\rho_{b\,exp}}$	(2)
	$\phi_b = \frac{X_b/\rho_b}{X_p/\rho_p + X_b/\rho_{b\,exp}}$	(3)
Specific heat	$C_{p_c} = \left[C_{p_{b\,exp}} X_b + C_{p_p} X_p\right] * \left[1 + A * X_b X_p\right]$	(4)
Thermal conductivity	$1-\phi_p = \left(\frac{\lambda_p - \lambda_c}{\lambda_p - \lambda_{b\,exp}}\right)\left(\frac{\lambda_b}{\lambda_c}\right)^{1/3}$	(5)
Viscosity	$\eta_c = \frac{\eta_{b\,exp}}{\left[1 - \frac{\phi_c}{\phi_{max}}\right]^2}$	(6)
	$\eta_c = \frac{\eta_0}{1 + \left(\frac{\eta_0 \gamma}{\tau^*}\right)^{1-n}}$	(7)
	$\eta_0 = D_1 exp\left[-\frac{A_1(T-T^*)}{A_2 + (T-T^*)}\right]$	(8)
Specific volume	$\upsilon_c = X_p \upsilon_p + \upsilon_{b\,exp}\left(1 - X_p\right)$	(9)
	$\upsilon(T,p) = \upsilon_o(T)\left[1 - Cln\left(1 + \frac{p}{B(T)}\right) + \upsilon_t(T,p)\right]$	(10)
	for $T > T_t$	
	$\upsilon_o = b_{1m} + b_{2m}(T - b_5)$; $B(T) = b_{3m} e^{[-b_{4m}(T-b_5)]}$;	(11)
	for $T < T_t$; $\upsilon_t(T,p) = 0$	
	$\upsilon_o = b_{1s} + b_{2s}(T - b_5)$; $B(T) = b_{2s} e^{[-b_{4s}(T-b_5]}$;	(12)
	$T_t(p) = b_5 + b_6(p)$ $\upsilon_t(T,p) = b_7 e^{[b_8(T-b_5)-(b_9 p)]}$	(13)

To perform mold-filling simulations, the tensile bar geometry model was imported into the Autodesk Moldflow software. The tensile bar geometry was meshed using an automated solid 3D meshing protocol based on finite element analysis. A gate size of 6.5 mm was set to perform molding simulations. A fill-and-pack type process module was selected to perform