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Influence of capillary die geometry on wall slip of highly filled powder injection molding compounds

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ABSTRACT

Uneven distribution of solid particles contained in the feedstocks used in the process of powder injection molding (PIM) is observed in the close vicinity of the walls. A particle-free thin layer adjacent to the walls is formed by the binder only and is characterized by so-called wall slip. Wall slip is a key to successful modeling of injection molding step of PIM. For its determination we used capillary rheometers equipped with the dies of different entrance angles applied to four PIM feedstocks. The entrance angle has been found to be a crucial parameter to intercept wall slip. Conical dies are more suitable to obtain reliable slip velocity values of highly filled compounds than capillaries having plane entrance, which are used in the majority of studies.

1. Introduction

The process of powder injection molding (PIM) introduces the material flexibility of powder metallurgy to the traditional plastic injection molding. The process involves four consecutive steps: incorporation of the metal (nano-, micro-) powder into organic binder, injection molding for shaping the component, removing (debinding) the binder from the component, and sintering that consolidates the final component [1].

Each step is of the same importance as the defects occurring in the previous steps cannot be eliminated in the subsequent steps thus terminating in appearance of cracks, internal stresses, dimensional distortion (warpage), etc. [2]. Therefore, the process starts with a choice of appropriate feedstock. Ahn et al. [3] demonstrate that binder selection is more critical than powder selection. German [4] and Li et al. [5] recommend to use a binder which viscosity does not exceed 0.1 Pa.s thus ensuring moderate viscosity (<1000 Pa·s) of the feedstock under the shear rates applied during injection molding. According to Fang et al. [6] only particles smaller than 20 μm are suitable for PIM, and finer powders are beneficial to improve the homogeneity. Every feedstock should exhibit shear thinning, i.e. viscosity decreases under shear strain. Nevertheless, a rate of shear thinning is a crucial point as e.g. for higher rates a cavity filling of complicated geometry requires less energy, but on the other hand, it can facilitate powder-binder separation [7].

Characterization of the powder-binder system is the introductory essential approach in modeling the PIM process complicated by the fact that the composition of PIM feedstocks typically range from 45 to 75 vol% filler content. Kate et al. [8] summarized empirical models for a prediction of a series of feedstock properties including their comparison with literature data on powder-polymer mixtures. They also presented the models for evaluating feedstock viscosity.

Rheological characterization gets more complicated during the second step: injection molding. This justifies an increased attention to a description of rheological behavior of the feedstock during injection molding as this characterization serves as one of the entries. Rheology of the feedstocks during injection molding was analyzed e.g. by [9–17].

As trial-and-error experiments are time-consuming and cost demanding, the computer simulation procedures not requiring performing the entire PIM process were proposed (e.g. [9,18,19]). The modeling of injection molding process with various molding parameters and complex material characterization was carried out by He et al. [20]. The simulations were in a good accordance with the experimental observations, however measured powder concentration within injected feedstocks was approximately 5–6% lower than simulated.

Such differences may be caused by no-slip conditions set in the most of models, while during real processing a contact between feedstock and channel walls results in the occurrence of so called slip layer (thickness attaining approximately 1/14–1/25 of a capillary diameter, details in [10,11]), containing only the pure binder system while

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the feedstock (with viscosity by orders higher) as a bulk material occupies most of the flow region [21,22], therefore special emphasis is necessary to pay to slip phenomenon that is the most notable rheological characteristic.

It was found [23] that no-slip condition leads to inaccurate simulation even in a simple pipe flow. In this respect, a method to determine wall slip layer thickness instead wall slip velocity was proposed [23]. Nevertheless, the wall slip correction model is still used solely if micro injection molding is simulated, because the wall slip is well pronounced in small flow channels. Choi and Kim [24] have shown that an implementation of a wall slip into flow simulations is necessary for channel diameters <10 μm.

Geometrical arrangement of the capillary dies participates significantly in the obtained rheological characteristics. An application of flat or conical dies changes both slip layer thickness and slip velocity. No obstacles as flat dies are encountered in the PIM process and rather smooth injection molding is met. The literature comparing flat and conical dies in connection with wall slip effect is rather scarce. Liang [25] and Ardakani et al. [26] tested changes in pressure drop with different capillary angles during extrusion. It was found that under constant pressure, shear rate increased with higher capillary entrance angle. The aim of this contribution is to compare rheological characterization of feedstocks obtained for both geometries of a capillary die and to show that a usage of the conical die is in better compliance with the analyses presented in the literature.

2. Experimental

2.1. Materials

Four types of commercially available PIM feedstocks (abbreviated P316L, P17-4PH, C316L, and C17-4PH, where P and C stands for two different binders, were used. The feedstocks are based on gas atomized spherical shaped stainless steel 316L and 17-4PH particles with sintered densities 7.75 g/cm^3 and 7.65 g/cm^3 for the binder P, respectively (P 316L , P 17-4PH), and 7.96 g/cm^3 and 7.67 g/cm^3 for the binder C, respectively (C316L, C17-4PH). The chemical composition of powders is depicted in Table 1. The feedstocks were prepared from the master alloys and carbonyl iron powder (ratio 1:3) having distributions of powder sizes >D_{90} 26 μm/D_{50} 9 μm/D_{10} 3 μm, and >D_{90} 8 μm/D_{50} 5 μm/D_{10} 2 μm, respectively.

The transition temperatures characterizing both polymer binders were obtained using a differential scanning calorimeter Mettler Toledo DSC1 Star. Fig. 1 depicts the results obtained, after cooling the samples to 0 °C, from second heating scan at a rate of 10 °C/min in the temperature range from 0 to 250 °C in a nitrogen atmosphere.

2.2. Rheological investigation

Two capillary rheometers with the dies of different entrance angles were used for an evaluation of the wall slip effect (Fig. 2). A Goettfert RHEOGRAPH 50 rheometer was equipped with 3 flat dies (180° entrance angle) of the same length-to-diameter (L/D) ratio (20/2, 10/1 and 5/0.5 in mm), a Rosand RH 2000 was used with 2 conical dies (90° entrance angle) of the same aspect ratio (16/1 and 8/0.5 in mm).

In the following we use the traditional Mooney method for the determination of the wall slip velocities. This method is based on changing the surface-to-volume ratio of the capillary dies, in other words, on changing a length L and radius R of the dies with fixing their ratio. In brief, we outline an evaluation of the experimental data.

Table 1
Composition of 316L and 17-4PH stainless steel powders (in wt%).

Powder	Fe	C	Ni	Mn	Si	Cu	Mo	Cr
17-4PH	Balance	<0.07	3.0–5.0	<0.1	<1.0	3.0–5.0	–	–
316L	Balance	<0.03	10.0–14.0	<2.0	<1.0	–	2.0–3.0	16–18

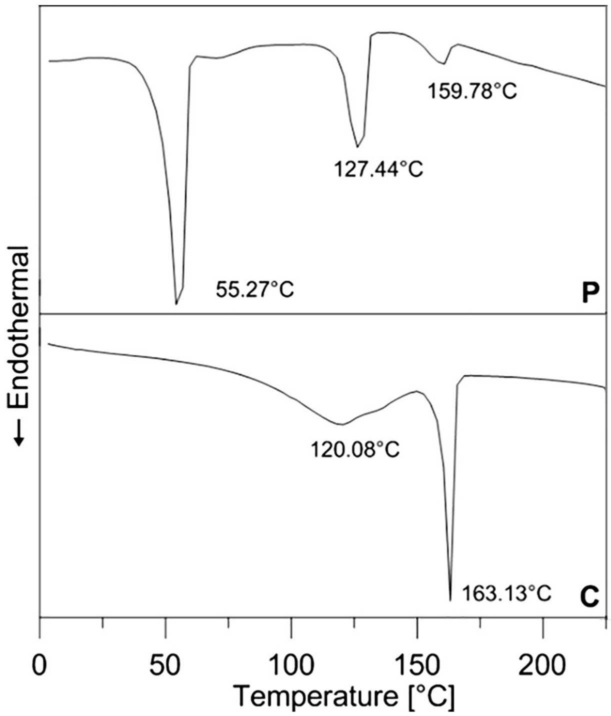

Fig. 1. Transition temperatures of both binders P and C (second heating scan at 10 °C/min in nitrogen atmosphere).

In a circular die an apparent wall shear rate $\dot{\gamma}_a$ and an apparent wall shear stress τ_a are determined by the relations

$$\dot{\gamma}_a = \frac{4\dot{Q}}{\pi R^3}; \tau_a = \frac{\Delta pR}{2L} \tag{1}$$

where $\dot{Q}$ is the volumetric flow rate and Δp is the measured pressure drop.

Consequently, a true average velocity is given by the difference of an average v_{av} $(=\dot{Q}/\pi R^2)$ and slip v_{slip} velocities

$$v_{true} = v_{av} - v_{slip} \tag{2}$$

Multiplying this relation by $4/R$ and using rel. (1) we obtain a dependence of the true apparent shear rate on the measured apparent shear rate and wall slip velocity

$$\dot{\gamma}_{a,\ slip-corrected} = \dot{\gamma}_a - \frac{4v_{slip}}{R} \tag{3}$$

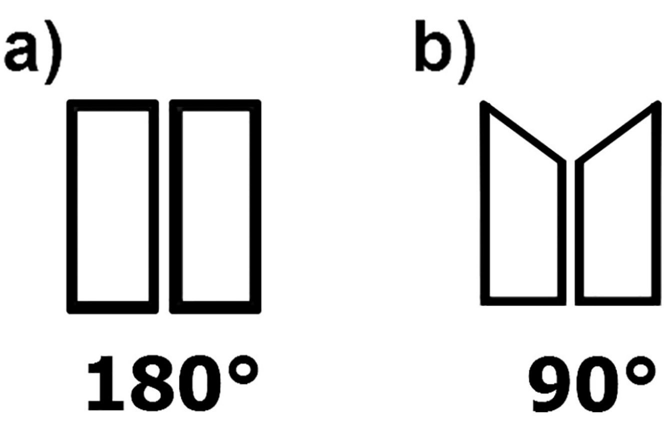

Fig. 2. Schematic representation of flat (a) and conical (b) testing capillaries.