Analysis of Feedstocks Separation during Powder Injection Molding

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Abstract

Separation of metal and ceramic powder particles from polymeric binder during powder injection molding (PIM) is the most frequent reason for quality failure of final sintered parts. Methods analysing the separation reported so far (X-ray, tomography, SEM) are limited to a qualitative description of this phenomenon. In this paper, Energy Dispersive Spectroscopy (EDS) of SEM of the testing samples derived from the specially designed injection mold are analysed with analytical computation approach providing quantitative, so called variability parameter, which depicts the susceptibility of the particular PIM feedstocks to the separation in the scale from 0 to 100 %. Then, non-destructive analysis of surface quality of the sintered parts determined with a contactless scanner with a First Interface Detection testing mode combined with suitable statistical methods allows comparing the tendency to the separation as a function of material composition and/or processing parameters.

1. Introduction

Powder Injection Molding (PIM) represents a processing route for metallic and ceramic powders that eliminates design restrictions inherent to conventional techniques. During the process the powder is mixed with a suitable polymer binder to obtain highly filled feedstock, which is then processed on injection molding machines utilized in plastics industry. In the next step, the polymer part is chemically or thermally withdrawn from the molded part prior to its sintering to the final density.

Although, PIM effectively combines plastic and metallurgical approaches gaining the benefits of both, the mass implementation of the production route is still limited by a number of issues.

Efficiency of PIM is to a great extent limited by material components’ separation during injection molding step. It originates from the high shear rates gradients located close to the walls of injection molding cavities. Resulting inhomogeneous redistribution of powder within a polymer binder can be evaluated with the help of specially developed testing mold allowing observing the progress of separation during injection molding \cite{1}.

In this contribution we focus on the testing method, recently developed \cite{2} for quantification of the separation for commercially available metallic feedstocks, on fine ceramic based feedstocks. The approach results in a single characteristic parameter, which helps to predict the structure defects as unacceptable porosity and/or cracks arising from injection molding affected by separation.
2. Discussion of Results

The scanning electron microscopy (SEM, VEGA II LMU, TESCAN) of the particular areas derived from the testing mold was combined with energy dispersive X-ray (EDX) analysis of the distribution of elements typical for powder (zirconium). SEM images of molded parts were taken at the magnifications 78, 94 and 115 according to the size of the particular square element of the testing mold, and accelerating voltage of 30 kV with BSE detector. Data collection for EDX was 20 times. The size of EDX maps were (2.8x2.8) mm on the first element, and then gradually decreased to (2.4x2.4) mm and (1.9x1.9) mm on the second and third elements, respectively. EDX quantification maps were derived with a resolution (128x128) pixels, representation of elements is expressed as weight % (wt%). SEM images (Fig.1 – left) and EDX maps (Fig. 1 – middle) of separated samples are shown below. Bright points represent powder and dark points represent binder. Also quantitative mapping was created ranging from 0 (black) depicting no Zr to 100 wt.% (white) showing the Zr appearance, Fig. 1 – right.

![Figure 1. SEM (left), EDX (middle) and quantitative EDX (right) maps of ZrO₂ feedstock at the first element of testing mold cavity](image)

Then, an analytical approach was employed to derive a quantitative parameter describing the tendency of highly filled feedstocks to the separation – a variability number. The rate of the phase separation represents non-uniformity of powder and binder distribution, i.e. non-uniformity of bright and dark points on EDX maps. The low and uniform variability anticipates an efficient PIM process without defects arising from the separation. The variation in zirconium is stable through the testing positions in the mold and reach the value 16±5 corresponding to fluctuations in the zirconium concentration about 70-75 %. The result is confronted with the surface appearance of the final parts (after polymer extraction and sintering) evaluated with the help of a contactless CLA surface scanner.

Acknowledgements

The Ministry of Education, Youth and Sports of the Czech Republic – Program NPU I (LO1504) is acknowledged for the financial support.

References