Mold Surface Analysis after Injection Molding of Highly Filled Polymeric Compounds

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This work deals with an impact of abrasive particles used in powder injection molding (PIM) on a surface roughness of the tool. For this purpose, the surface of new mold cavity was compared with the same mold cavity after 2 000 injection molding cycles. Processed PIM compounds contained polymeric binder with around 60 vol. % of metal or ceramic particles (0.1 up to 20 μm). Surface analysis was performed on cavity impressions prepared from a special silicone imprinting substance in two directions by a 3D surface scanner. Investigated parameters were surface roughness (Ra) and roughness depth (Rz) which have an influence on flow instabilities of highly filled compounds such as wall slip affecting the final product quality. Obtained results showed a significant wear of the mold cavity which was statistically confirmed by t-test and F-test parametric methods. A greater part of the mold cavity was smoothed during injection of PIM compounds, while the surface roughness increased near the point gate (runner system) probably due to a high injection pressure in this part of the mold.

Keywords: PIM, Cavity, Surface, Roughness, Wear

1 Introduction

Powder injection molding (PIM) is a relatively new technology capable of transforming complex concepts and design into high precision, outstanding final properties, and net-shaped products from a wide range of materials. PIM comprises four main steps: mixing, injection molding, debinding and sintering. Initially, ceramic or metal powder particles are mixed with a suitable polymeric binder system into a homogenous mixture. The binder system provides not only appropriate flow properties, but also holds the selected powder particles together prior to sintering. Then, the mixture called PIM feedstock is fed into injection machine (usually in a form of pellets), where it is injected in a molten state under pressure into the mold cavity. Injection molding is followed by debinding during which the polymeric binder is removed by various routes: thermally, by solvents or catalytic reaction or by their combination. Finally, in the last sintering step, the powder particles are bonded together until almost full density [1-3].

Injection molding is the last step in which the process is still reversible. Therefore, the quality of the tool, which is mold cavity in injection molding machine, is very important for final product. During injection under pressure, the inner surface of a mold cavity is exposed to a wear due to abrasive character of metal, and especially ceramic particles. Typical feedstock contains high amount (about 60 vol. %) of very fine powder particles, as it is shown in Fig. 1 obtained from scanning electron microscopy (SEM). Typical metal particles have size between 1 – 20 μm, while the ceramic particles are usually even finer. These particles are very abrasive, especially in case of ceramics, and thus the quality of mold surface could be changed during injection molding [1]. This can enhance flow instabilities of the feedstock such as wall slip [4] or it can cause the dimensional variation of the final PIM products.

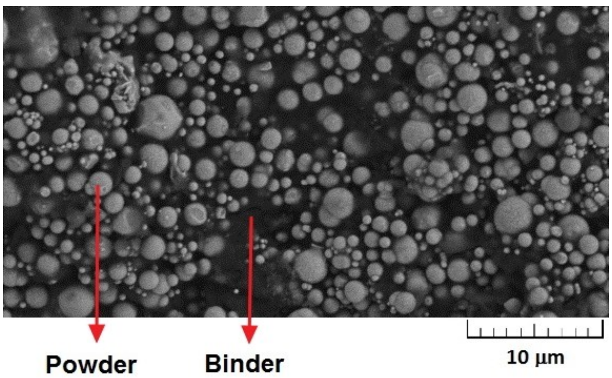

Fig. 1 *SEM micrograph of PIM feedstock*

The quality of the mold and its durability is important because even minor imperfections in shape and surface of the cavity can lead to a waste production [5]. The wear caused due to abrasive ceramic or metal particles in PIM feedstocks is significantly higher than the wear caused by injection molding of pure polymeric material. As the higher wear in PIM shortens the tool life, the understanding of the wear is very important. Especially regarding to the commercial use a reduction of the wear effects during PIM is essential. However, there is not enough research done in this field.

The aim of this research is to evaluate the surface quality of the cavity by means of average surface roughness (Ra) and roughness depth (Rz) after 2 000 injection cycles of various PIM feedstocks based on stainless steel (17-4 PH, 316 L) and ceramic (Al_2O_3, ZrO_2) powder particles. The surface roughness is one parameter which may indicate the quality of surface in the mold cavity. Direct measurement of surface roughness may be difficult or impossible due to the shape of the cavity, dimensions of the mold as well as its weight. That is why in this work, we try to use a special impression substance and create firstly the imprint of surface in the mold cavity and subsequently, we measure the topography of surface by a contactless method. The main purpose of this paper was to develop a new method for recording the data and characterizing the wear phenomena in PIM process without using tool inserts but directly in the existing mold cavity as well as the understanding of surface changes, which can prevent the flow instabilities during mold filing and help to design the mold.

2 Experimental

2.1 Mold cavity design

Investigated tool is a cavity in the injected mold especially designed and patented at Polymer Centre (Tomas Bata University in Zlin) in cooperation with Fraunhofer (IFAM, Bremen - Community Design 001704974-0001) in order to investigate the flow instabilities occurring at high shear deformation of highly filled polymer compounds during mold filling [6]. A schematic view of the testing specimen design intended for injection molding is shown in Fig. 2. This specimen contains four elements separated from each other; first three elements in the direction of the flow were investigated. All three elements have a square shape with the same inner side length of 10 mm and were designed to evaluate the alterations in a phase separation. The knowledge of surface roughness is very important for such type of testing mold, because during flow, the wear phenomena could lead to irreproducible flow instabilities as the powder contained in the injected PIM feedstocks causes changes in a surface roughness.

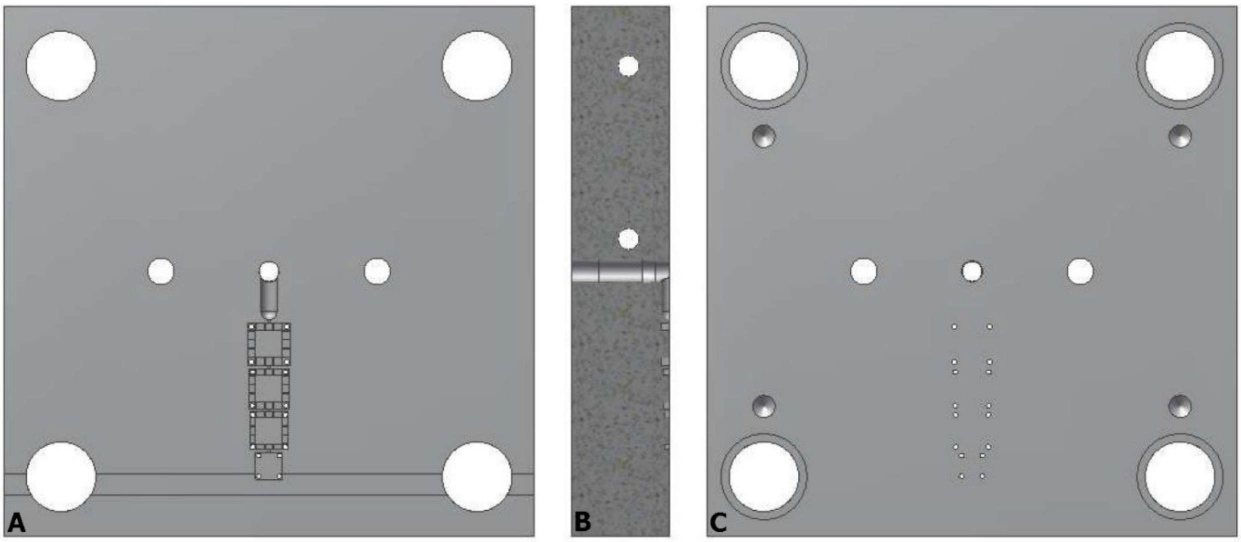

Fig. 2 *View of left cavity plate - a) front, b) cross-section, c) back* [7]

Left and right cavity plates with the size of (196 x 196 x 36) mm are made from a standard tool steel DIN 1.2767 - X 45 NiCrMo (ISO 4957:1999) which chemical composition is depicted in Tab. 1. Both cavity plates were hardened to 56 HRC by further heat treatment. This steel enables to be hardened with minimum distortion and also possesses good thermal properties for injection molding. It also displays excellent polish ability and toughness in the hardened condition making it suitable for high pressure injection molding applications. Contact surface of the cavity plates were polished to Ra 0.2 μm, and the cavity itself was made with higher roughness specifically for flow instabilities study.

Tab. 1 *Chemical composition (wt. %) of DIN 1.2767*

C	Si	Mn	P	S	Cr	Mo	Ni
0.4-0.5 ± 0.03	0.1-0.4 ± 0.03	0.2-0.5 ± 0.04	max 0.03 + 0.005	max 0.03 + 0.005	1.2-1.5 ± 0.07	0.15-0.35 ± 0.03	3.8-4.3 ± 0.07

2.2 Imprinting method

To allow cavity surface evaluation before and after 2 000 injection cycles, a special method of scanning negative imprints of mold cavity was adopted. Both imprints of the surface cavity were made by a special two components imprinting substance based on silicone Stomaflex® Light supplied by Spofa. This compound, combining a classical putty and low-viscosity light material, offers more precise detail reproduction, comfortable handling, good tolerances, health safety and an easy control of homogenously mixed material with color-contrast catalyst. The only drawback is the possibility of enclosing air. Fig. 3 demonstrates the placement of mold cavity imprint on the working table of the scanner for surface evaluation.

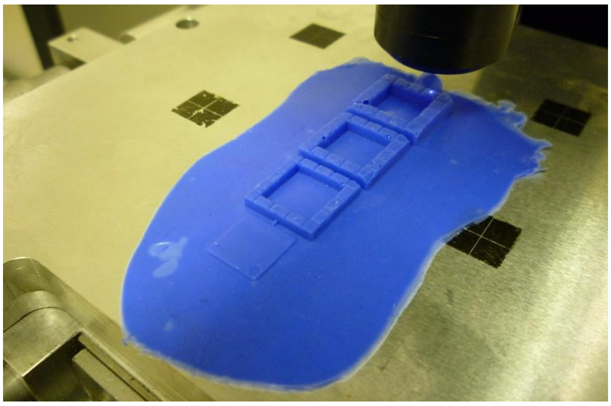

Fig. 3 *Imprint of mold cavity ready for scanning*

2.3 Imprint surface scanning

The specific surface areas of produced mold cavity imprints were scanned and evaluated by a 3D surface scanner Talysurf CLI 500 (Taylor Hobson, UK) using a Chromatic Length Aberration (CLA) technique. This method employs a polychromatic light beam with an error less than 20 nm and provides highly accurate non-contact 3D measurement offering the presentation of surface such as isometric view or topographic maps. Fig. 4 shows a topographic map of the mold's surface in 3D using a commercial software Talymap Taylor Hobson Company.

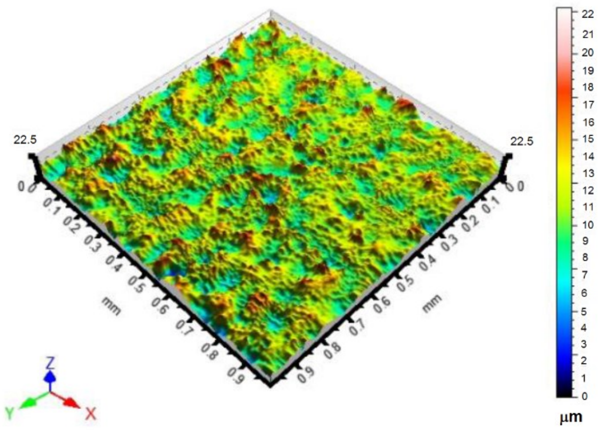

Fig. 4 *Scanned surface topography*

Scanned surface topography was evaluated from first three elements (in direction of flow) before and after 2 000 cycles in specific areas (analyzed space /1x1 mm/). This specific areas were analyzed in two directions (axes *x* and *y*), standard ISO 4287:1997 was used for measuring surface roughness (Ra), roughness depth (Rz) and standard ISO 4288:1998 for methodology. Measured values were mathematically processed using a software Minitab 14 into boxplot diagrams. Surface roughness is the most common parameter for determining the quality of surface topography; it is an arithmetical average of the sums of all profile values from the mean line. The other parametr, roughness depth is the maximum height of the roughness profile which is calculated as a sum from the height of the highest profile peak and depth of the lowest profile valley within a sampling leght [8].

3 Results

The arithmetical averages of measured surface roughness (Ra) and roughness depth (Rz) have been performed at twelve different places in both directions. Obtained data was then statistically evaluated and in all cases the same trend was found, therefore Fig. 5 demonstrates boxplot diagrams only for Ra parameter measured in *x* direction, where the measured places were labelled in each specimen part (as A, B, C, D), while (0) indicated data before and (1) after 2 000 injection cycles. As it can be seen, the diagrams clearly show reduction of the surface roughness, the only exception was in position 1A and 1B where this trend was not confirmed.

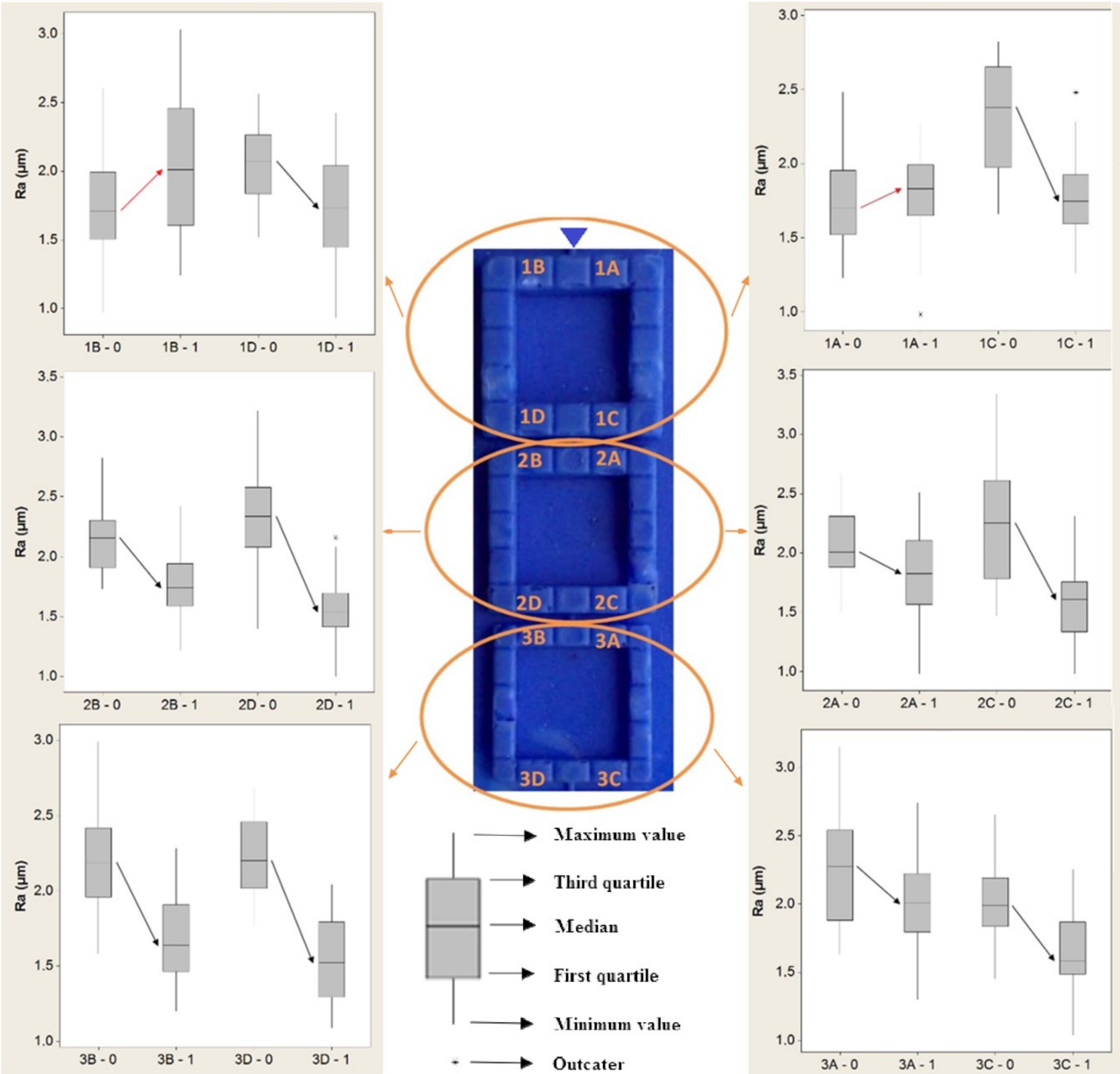

Fig. 5 *Boxplot diagrams of measured Ra in x - direction*

In order to determine if this trend of surface smoothing is not only coincidence in the measurements, the statistical evaluation, specifically t-test and F-test for a confidence level of 95 %, was carried out. According to the statistical Anderson-Darling test (test of normality) for above established confidential level, it can be said that all Ra and Rz values belong to Gaussian (normal) distribution set of parameters N (μ, σ^2). T-test rejected in all cases the null hypothesis (H_0: $\mu_0 = \mu_1$) except of position 1A, while the F-test null hypothesis (H_0: $\sigma_0^2 = \sigma_1^2$) was rejected six times for Ra and twice for Rz parameter (besides both measured directions). Based on the above results (with coefficients of variation between 12 - 20 %), it can be concluded that a significant wear of the mold cavity was statistically confirmed by parametric methods (t-test and F-test) with 95% confidence level.

The opposite trend in the position 1A and 1B can be explained due to higher injection pressure in the first part of mold, where it forms the cavity effect. In order to investigate pressure evolution in the mold cavity during injection, the flow simulation was performed with the help of Moldflow 2014 software. The analysis was set using a dual domain mesh with 0.45 mm edge length and 48 220 triangular elements and default processing parameters recommended for Zirconia compound PXA (Tosoh Corporation) due to only comparative character. As it can be seen in Fig. 6, this effect may be caused by higher injection pressure, and also by unstable flow at the entering the mold cavity.

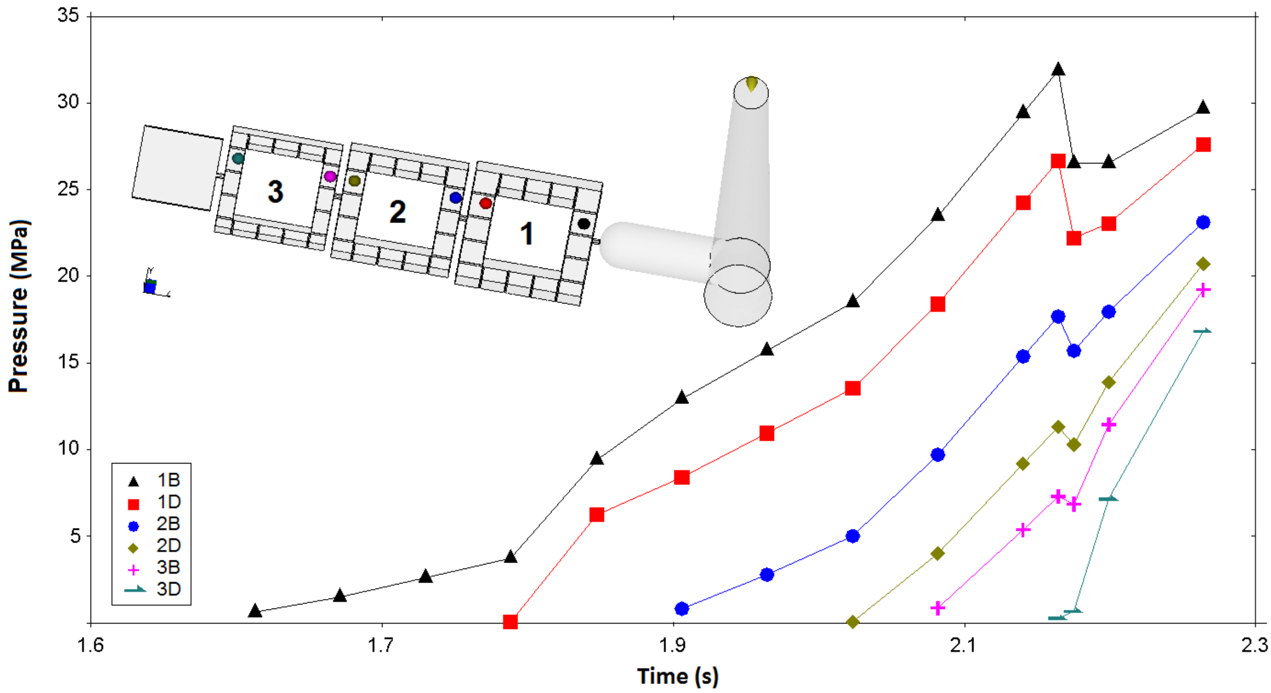

Fig. 6 *Tendency of mold cavity pressure*

4 Conclusion

The possibility of surface profile measurement by 3D scanner provided very useful information about the geometric characteristics of the replicated cavity surface using a special impression material. This means that 3D surface evaluation opens a new opportunities for a comprehensive evaluation of the surface quality not only in PIM technology. From the experimental evaluation, it can be concluded that there is a significant change of the surface roughness on the examined places in the mold cavity after 2 000 injection cycles, which was statistically confirmed by parametric methods (t-test and F-test) with 95 % confidence level. The changes of surface must be taken into the account during flow instability evaluation as well as design a mold and injection molding step of PIM, because the quality of mold surface affects not only the final structure of metal or ceramic products, but also the homogeneity of feedstock after injection molding.

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