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Analysis of Shrinkage in Metal Injection Molding

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ABSTRACT

Maintaining component dimensions in metal injection molding (MIM) is difficult because of significant component shrinkage in the sintering step and shrinkage variation. Most of the shrinkage variation in MIM is due to density inhomogeneity in moldings (greens), induced in the injection molding step. Therefore, the focus of this research was to clarify the relationship between injection molding parameters and shrinkage of a ring-shaped component molded into a four-cavity split mold. Material used in research was Catamold 310N in the form of ready to mold granules. An experiment, according to a 2k factorial design, was performed to evaluate the effects of holding pressure profiling on shrinkage at different levels of coolant temperatures and fill rates. Results showed that the interaction of holding profile and coolant temperature has the most influence on the mean shrinkage. This finding offers a possibility to maintain the component dimensions in MIM by using the revealed interconnection between holding pressure profile and mean shrinkage.

Key words: *metal injection molding; shrinkage; holding pressure profile*

1. INTRODUCTION

Metal injection molding (MIM) is a technology for net shape production of small and complex metallic components. The major technological steps in MIM, when producers rely on a commercial granulated mix of metallic powder and binder (feedstock) are:

- injection molding, where melted feedstock is transferred, pressurized and cooled in the mold cavities forming a so-called green component,
- debinding, where most of the binder is removed from the green component to get a shaped porous and dominantly metallic part,
- sintering, where the porosity and the part dimensions are significantly reduced to achieve metallic parts with the required density.

Maintaining component dimensions in the typical narrow tolerance range of $\pm 0.3\%$ is very difficult because of significant nominal shrinkage of 12% to 18% during the sintering step and inevitable shrinkage variation. The nominal value of shrinkage depends on the feedstock powder-binder ratio (solid loading) and final component density, while the shrinkage variation is related to: row material variation, cavity machining and processing

variation. The row variation of the feedstock (batch to batch variation) is induced in the preparation (mixing and granulating) phase, and is unavoidable when MIM producers are relying on commercial feedstock. This variation with the machining error of cavities often consumes almost the entire tolerance budget in MIM. There is also the shrinkage variation that appears during the processing, a so-called within batch variation. This variation is mostly induced in the injection molding step, where the initial homogeneity of feedstock is reestablished during melting, mixing and filling sequences, resulting in unavoidable density gradients in the green's [1,2]. Green density variation and the gravity effect are mean causes of anisotropic (non-uniform) shrinkage during subsequent debinding and sintering steps [3,4]. Moreover, non-symmetrical temperature and viscosity distributions in the runner system of multi-cavity molds resulted in a filling imbalance and cavity to cavity shrinkage variation. This filling imbalance is influenced by the runner's geometry, the thermal and rheological characteristics of the feedstock and the injection molding parameters [5]. To accommodate non-uniform shrinkage, producers make sure that the tool cavities can be reworked after first sampling experiments. Additionally,

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to maintain a narrow tolerance range, MIM producers introduce post-sintering operations: machining or operations to increase component density.

An additional possibility to achieve tolerances in MIM, for producers using commercial feedstock, is to control the shrinkage mean and variation by using injection molding parameters. But, finding an interconnection between processing parameters and shrinkage requires deep understanding of the MIM process and complex thermo-rheological properties of the MIM feedstock. A review of the literature has shown that the relationship between injection molding parameters and shrinkage in multi-cavity molds is an insufficiently explored area. Therefore, the aim of this research is to clarify the effects of holding pressure, fill rate and mold temperature on shrinkage of a complex ring-shaped MIM component using designed experiments.

2. EXPERIMENTAL WORK

2.1 Machines and components

The Arburg 320C injection molding machine adopted for the MIM process was used in injection experiments. This machine controller offers options for profiling injection velocity and holding pressure in time. An experimental four-cavity split mold was designed for the molding experiments, Fig. 1 (right). The cavities are filled through a vertical runner system with pinpoint gates, as depicted in Fig. 1 (left).



Fig. 1 CAD model of vertical runner system and components (left); mold cavities between two splits of mold (right)

In this study, a small complex ring-shaped component (mass 1.27 g) with an external groove in the middle, was analysed. The cross-section of the component is shown in Fig. 2 (left), oriented as standing on debinding and sintering steps. Catalytic binder degradation was done in an Elnik 3002 CE oven in a nitric acid rich atmosphere, according to BASF process. After debinding, the brown parts were carried to the Elnik batch furnace MIM 3002 where sintering at 1300°C in three hours using N₂ atmosphere was performed. Measurements of dimensions were performed using DEA Global CM Machine with specified accuracy MPE-E 2.1+1L/333 [µm].



Fig. 2 Component cross-section oriented as standing during sintering (left); runner system, green and coponents after sintering (right)

2.2 Material

Material used in this research was Catamold 310N in the form of granules made of heat-resistant stainless steel X40CrNiSi 25-20 EN powder and polyacetal (POM) based binder. Catamold 310N is highly viscous where viscosity is mainly influenced by shear (rate), when it is processed in a temperature range from 170 °C to 190 °C. The POM binder has a high crystallinity (70% to 80 %) with the crystallization temperature of 140 °C. The oversizing factor (k = tool dimension/ dimension after sintering) of Catamold 310N based on long term batch to batch variation is reported as k =1.1669 ± 0.004. This value corresponds to the linear shrinkage of 14.3 ± 0.3 %, see Fig. 3.



Fig. 3 Catamold 310N batch to batch variation of oversizing factor according to BASF

2.3 Variables choice

Review of the literature has shown that the injection molding parameters: holding pressure, mold temperature, fill rate, melt temperature and also feedstock parameters: solid loading and homogeneity, have major effects on shrinkage. Tseng (1998) is established that the holding pressure and the interaction between holding pressure and mold temperature are parameters that have the greatest influence on shrinkage deviation from the nominal dimensions of the cavity. Green, C. D. et al. (2007) proved that the injection velocity and the holding pressure have an influence on the component dimensions after sintering. Prathabrao M. et al. (2018) showed that the melt temperature has an effect on the viscosity, and consequently on the ability of the melt to fill up the cavity. Quinard et al. (2009) showed that the feedstock homogeneity minimize powder-binder helps to

segregation during the injection molding stage and to obtain isotropic shrinkage. Husnareva, B. et al. (2006) established that the increased metallic powder concentration (solid loading) in feedstocks diminishes the pressure sensitivity and shifts the feedstock crystallization point toward lower temperatures. Bulger M. K. et al. (1997) showed that the solid loading lowering increases the effect of gravity, which plays a clear, but not always primary, role in anisotropic shrinkage. Parameters: coolant temperature (T), holding pressure profile (P) and fill rate (v) were chosen in the study, based on previous investigations, material producer recommendations and experience. The fill rate was profiled: first the runner system was filled at a rate of 10 cm³/s, then the fill rate was reduced and varied in the range of 6 cm^3/s to 7 cm^3/s , up to switchover point. The processing window of holding pressures was searched using a constant fill rate of 10 cm³/s and the criteria "green parts without defects" (flash, underfill, cracks). Finally, two holding pressure profiles were chosen: first with a linear decrease from 85 MPa to 80 MPa (rump-down), and second with a linear increase from 85 MPa to 90 MPa (rump-up), both in rump time of 2.1 s. In order to examine the effect of mold temperature on feedstock ability to fill cavities, two coolant temperatures were chosen: 115 °C and 125 °C. A list of control variables with variation ranges and variables held constant in this study is shown in Table 1.

Table 1 List of variables an	d levels used in the e	xperiment
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Control variable	(unit)	Level 1	Level 2
v - fill rate	cm ³ /s	6	7
P - holding pressure profile	MPa	85-80	85-90
T - coolant temperature	°C	115	125
Held constant variable			
E - melt temperature	°C	185	
F - packing time	s	2.1	
G - cooling time	S	16	-

3. RESULTS AND DISCUSSION

Experiments were conducted according to the (2^4) design matrix shown in Table 2. For all combinations of control variables, three green components from the upper (1) and three from the lower (2) cavity were taken for measurement. Measurements of the component height (H), inner (ID) and outer (OD) diameter were carried out on the greens and on the components after sintering, see Fig. 2, then the linear shrinkages were calculated. To identify shrinkage variation between cavities, the vertical position of the cavity in the mold (JJ) was used as a blocking variable.



Fig. 4 Comparative box plots of shrinkage variation for observed component dimensions

Preliminary results showed that variation of input parameters, according to design matrix, produce similar shrinkage variability of observed component dimensions, as seen in Fig. 4. Tests of equality of the means showed a significant difference in average shrinkage between H and ID (diff. 0.18%; p-value=0.022) and between H and OD(difference 0.28%; p-value=0.000), at 95% confidence. The gravity effect in the sintering phase could explain significantly higher shrinkage in the H direction.

Table 2 - Design matrix, linear shrinkage of component dimensions and shrinkage standard deviation

					L	Shrinkage		
	Exp	. V P	T JJ		[(lg-ls)/lg]x100 [%]			SDev
		cm ³ /s M	Pa ⁰ C	-	OD	ID	Н	[%]
1	6	80	115	1	13.8518	14.1443	14.5524	0.3519
2	7	80	115	1	14.1964	13.7566	13.9347	0.2212
3	6	80	115	2	14.0016	13.8036	14.0749	0.1404
4	7	80	115	2	13.9456	14.0143	14.3232	0.2011
5	6	90	115	1	14.4566	14.0485	14.0956	0.2233
6	7	90	115	1	14.5124	14.2989	14.5324	0.1294
7	6	90	115	2	14.1066	14.3439	14.6295	0.2618
8	7	90	115	2	14.1924	13.9649	14.2456	0.1491
9	6	80	125	1	14.5790	14.1125	14.6048	0.2771
10	7	80	125	1	14.3786	14.2538	14.4727	0.1098
11	6	80	125	2	14.0801	14.2406	14.5954	0.2637
12	7	80	125	2	14.0094	14.0882	14.4630	0.2424
13	6	90	125	1	14.3786	14.0048	14.1632	0.1876
14	7	90	125	1	14.1851	14.3007	14.4677	0.1421
15	6	90	125	2	14.1103	14.0503	14.2807	0.1195
16	7	90	125	2	13.9231	13.9209	14.4456	0.3023

The Minitab ANOVA was performed to reveal the influence of input variables on shrinkage variation between observed component dimensions. The main effects for shrinkage standard deviation showed that high levels of fill rate (v) and rump-up holding pressure profile (p) could be used to minimize anisotropy in shrinkage, see Fig. 5. Higher fill rates followed by higher holding pressures lead to higher pressure in cavities, better packing and density homogenization, and consequently lower shrinkage anisotropy as expected.



The Minitab ANOVA for the linear shrinkage as response variable was performed to reveal injection parameters influencing the shrinkage mean. As shown from the ANOVA report, see Table 3, the variation between cavities (*JJ*) contributes to total variation with 4.8 %, while between dimensions variation (*AN*) contributes with 26 %.

Table 3 - Minitab Analysis of Variance for the shrinkage, usingAdjusted SS for Tests

Source	DF	Adj SS	Adj MS	F	Р
v	1	0.00487	0.00487	0.15	0.699
Т	1	0.09034	0.09034	2.82	0.102
Р	1	0.02905	0.02905	0.91	0.347
T*P	1	0.41720	0.41720	13.02	0.001
T*V	1	0.00021	0.00021	0.01	0.936
P*V	1	0.02640	0.02640	0.82	0.370
T*P*V	1	0.00476	0.00476	0.15	0.702
JJ	1	0.12286	0.12286	3.83	0.058
AN	2	0.66338	0.33169	10.35	0.000
Error	37	1.18581	0.03205		
Total	47	2.54487			

The shrinkage variation between cavities obtained is a clear sign of an imbalance in filling. A confirmation of this is a higher percent of binder mass in the green pressed in the upper cavity, evaluated as mass difference between green and components after sintering. This could be explained by the underpacking effect, where more volume of the upper cavity was filled under lower holding pressure after V/P switchover point. Possible reasons for the cavity to cavity variation obtained are: temperature imbalance caused by serial pattern of cooling channels, gravity influence due to difference in vertical position of

cavities and mold machining errors.

As for injection molding parameters, their contribution to total variation is 22 %. The F-test showed that only the main effect of T^*P interaction has a statistically significant influence on mean shrinkage, with a p-value of 0.001, Table 3. Interaction plot of T^*P , see Fig. 6, shows that the relationship between holding pressure profile and shrinkage changes direction based on coolant temperature. This phenomenon could be explained by the feedstock sensitivity to temperature and pressure. Namely, when the coolant temperature is lower, owing to thermal conductivity of MIM feedstock, high crystallization on the colder runner walls starts almost immediately. Therefore, an earlier gate sealing and consequently a lower packing, could explain comparatively lower shrinkage on average at a lower level of coolant temperature (115 °C). As for the holding pressure influence, generally, higher pressures cause an increase in pressure in the cavities, that leads to more packing and decrease in shrinkage. In our experiments, at a coolant temperature of 125 °C, the pressure increased according to the rump-up pressure profile, leading to a decrease in average shrinkage of 0.13%, as expected. But, at a coolant temperature of 115 °C, the change from rump-down to rump-up pressure profile caused an increase in shrinkage of 0.27 %, quite opposite to expectations. A possible explanation for the contrasting results obtained lies in feedstock sensitivity to pressure. Here a lower coolant temperature promotes melt crystallization on the walls of the runner system, reducing their cross-sections, increasing melt velocity and also melt temperature due to shear heating.



Fig. 6 Interaction plot for P*T, Average shrinkage variation of the components from both cavities pressed with the fill rates 6 and 7 cm³/s

This shear heating and increase in temperature will reduce feedstock solid loading, since the thermal expansion coefficient of the POM binder is about one order higher than steel powder. Such a feedstock becomes pressure-sensitive, where the pressure increase shifts the crystallization temperature toward higher values [12]. The gates sealing at higher melt temperatures and consequently lower packing could explain the shrinkage increase of 0.24 % when the rump-up (85-90 MPa) pressure profile was used.

3. CONCLUSION

One particularly interesting finding of research is that MIM feedstock sensitivity to temperature and pressure could be used to affect quality in MIM. Experimental results showed that an increase in holding pressure (5 MPa in 2.1 s according to rump up profile) resulted in an expected negative relationship between holding pressure and mean shrinkage at coolant temperature of 125 °C. But the same increase in holding pressure, at coolant temperature of 115 °C, reversed this negative relationship. This is a consequence of pressure-sensitivity of feedstock with lower solid loading, where the pressure increase causes an increase in crystallization temperature. Affecting melt crystallization by pressure profiling (increasing or decreasing) offers the possibility to control component shrinkage and dimensions in MIM. This approach could be used for adjusting the shrinkage factor for every new batch to compensate for inevitable batch to batch shrinkage variation.

It would be advisable to conduct additional research to explore potential non-linear interdependence by changing influential factors at three or more levels.

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