

A methodology for shrinkage measurement in micro-injection moulding

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Abstract. The aim of this paper is to provide a method for measuring shrinkage in micro injection moulded (μ -IM) parts – no standardised approach being reported as yet in the literature. This study investigates the feasibility of implementing the international standards used to investigate shrinkage in conventional (macro) moulding at the micro-scale. Following a similar experimental procedure to the relevant standards, micro-moulded polyoxymethylene (POM) specimens were produced, and the influence of processing parameters on their shrinkage was analysed using the design of experiment (DoE) approach. The analysis results showed that the methodology was capable of detecting factors that had a statistically significant effect on shrinkage at the micro-scale, in both parallel to, and normal to, the flow directions for moulding, post-moulding and total shrinkage.

Keywords: micro injection moulding; shrinkage; design of experiment.

1. Introduction

1.1 Scale effects and experimental measurement of shrinkage in micro-scale

There appears to be no work in the research literature that compares shrinkage behaviour on the micro-scale to that observed on the conventional macro-scale for a particular feedstock material during injection moulding. Moreover, some studies have highlighted the possibility of scale-induced differences in flow behaviour, which may affect the shrinkage induced during injection moulding.

For example, Liu et al. recently studied the morphology in the flow direction of both macro and micro parts (0.2 mm in thickness), moulded from isotactic polypropylene (iPP) [1]. They found that the through-thickness morphology of micro-parts exhibited a similar “skin core” type of structure to the macro-parts.

However, in the micro-parts a large fraction of shear layer was present, whereas the macro-parts presented a large fraction of core layer. The shear layer of micro-parts had a highly oriented ‘shish kebab’ structure, with a pronounced orientation of iPP chains within lamellae. The authors calculated that the percentage thickness of the oriented region of the micro-parts (which included both the skin layer and the shear layer) was much greater than in the macro-parts (90% versus 15%). These different degrees of orientation observed at the macro and micro-scale, indicate that shrinkage data obtained at the macro-scale is unlikely to be directly extrapolatable to the micro-scale. They indicate the necessity of directionally dependent measures of shrinkage for micro-scale components, as the orientation is likely to lead to observable anisotropy in the shrinkage behaviour. Furthermore, results have been previously reported for POM indicating that crystal size is related to moulding scale, with smaller mouldings containing finer crystals – an effect attributed to rising thermal nucleation owing to the increased cooling rate usually observed at the micro-scale [2, 3].

1.2 Micro-scale shrinkage measurement

Two papers have considered a non standardised approach to a measurement shrinkage methodology. Règnier et al. manufactured a square mould with triangular channels, 30 µm deep, 50 µm wide and 12 mm length [4]. Their results indicated that injection time and the specimen thickness have some influence on shrinkage. In the work of Lee et al. [5], the authors realized equipment for determining the shrinkage, but no measurements were performed.

To date, only one study has adapted a widely used standard to the micro-scale [6]. The mould and the specimen followed the standard macro-scale reference ASTM D955-89 [7], i.e. a rectangular bar of length to width ratio of 10:1. This was a continuation of work at the macro-scale [8]. The authors determined the amount of shrinkage in three commercial polymers: acrylonitrile butadiene styrene (ABS), polystyrene (PS) and high density polyethylene (HDPE) [6]. For the micro-scale experiments the specimen dimensions were reduced scale by a factor of approximately 25 as compared to the ASTM D955-89 standard.

1.3 Purpose of paper

The aim of this paper is to provide a method for measuring shrinkage in micro-moulded parts. The term “micro” indicates that one or more dimensions are sub-millimetre in size [9].

For the study three standards are cited:

ASTM D955-89 [7] describes three different moulds for measuring the shrinkage (rectangular, square, circular). The recommendation is to use a square specimen for determining shrinkage parallel and normal to the flow;

ISO 294-3 [10] gives the square mould dimensions for macro-scale. The corresponding mould has overall dimensions of 180 mm x 72 mm (figure 1);

ISO 294-4 [11] reports the mathematical formulas and the measurement methodology for measuring the shrinkages (mould, post-mould and total shrinkage). Standard ISO 294-4 is currently used for measuring the dimensional variations in macro-scale injection moulding, both in the technical datasheets of material suppliers and more sparsely in technical publications [12-14].

In previous work at the micro-scale, described above, a rectangular mould was used, with an edge gate, but shrinkage was measured both parallel to and normal to the flow [6]. However, ASTM D955-89 recommends a square specimen when shrinkage normal to the flow is to be measured.

Therefore, the approach taken here was to implement a scaled-down version of the square mold design implement in ISO 294-3 [10]. Such a design would also comply with the square mould design from ASTM D955-89. In the present paper, the steps of adopting this methodology will be described. As a validation of the methodology, results of experiments to determine whether there any statistically significant moulding parameter effects on shrinkage are presented. These are compared with the small amount of prior data available in the literature.

2. Methodology

2.1 Calculating shrinkage

The square mould design is depicted in figure 1. Table 1 reports the mathematical formulas for determining the shrinkage as indicated in [11]. The shrinkage is expressed as percentage related to the cavity mould dimension. The parameters reported in table 1, are defined as follows:

S_M (moulding shrinkage) is the difference in dimensions between a dry test specimen after 1 hour and the mould cavity in which it was moulded;

S_P (post moulding shrinkage) is the difference in dimensions of moulded test specimen after 1 hour and 24 hours;

S_T (total shrinkage) is the difference in dimensions between a test specimen after 24 hours and the mould cavity in which it was moulded.

2.2 Adapting the macro standard to the micro-scale

The design of micro-mould maintained the general shape (triangular runner, square specimen, linear dimension/specimen thickness ratio) stipulated in ISO 294-3 [10].

The triangular gate design stipulated in ISO 294-3 (figure 1) was followed. This design should create a uniform melt flow without turbulence, to allow a wide polymer front for filling the cavity mould,

minimising melt fracture. The triangular gate permits the creation of a uniform polymer flow for filling the mould cavity, because the edge gate [15] generates an elliptical melt front. Triangular gates have been used in models of the polymer filling process in micro channels [16] and for determining the orthotropic shrinkage in conventional moulding [4].

Figure 2 depicts a “no-shrinkage” specimen, which scales down the stipulated ISO 294-3 mould dimensions (length range: 60 to 62 mm; height range: 2.0 mm to 2.1 mm) by a factor of six to be suitable to the micro-scale.

2.3 Mould manufacture

The micro-mould for this specimen design was realized by micro-milling machining with a precision of $\pm 1\mu\text{m}$. A HLS 2003-020 tool was used for manufacturing the mould, with a diameter of 300 μm , effective length of 2 mm and a flute length of 0.4 mm. The rotational speed was approximately 30,000 rpm and the feed rate was of 45 mm min^{-1} . An oil/air coolant was used. After micro-machining, the final dimensions of the single square cavity were length= 9.987 ± 0.001 mm, breadth= 9.980 ± 0.001 mm, height= 0.350 ± 0.001 mm. These values were used as the reference for calculating the shrinkage results reported in table 5. Figure 3 depicts the mould ejector pin size and placement. Eight 1 mm thick ejector pins, symmetrically positioned were used in the micro-mould design to reduce the likelihood of the bending the thin specimen on ejection.

2.4 Processing Parameters

Processing parameters, material and external influences can affect shrinkage. The present paper investigated the processing parameters influences, whilst keeping the material constant. Five factors were adopted in the present study (table 2). These parameters have been noted in prior work to affect macro-scale shrinkage [17]. The same processing parameters have been chosen for non-shrinkage micro-moulding studies by several research groups, [18] [19] [20]. Table 2 also indicates the change in shrinkage noted at the macro-scale on increase of the magnitude of a processing parameter [17]. For each of the processing parameters listed in table 2, a preliminary experimental screening was used to identify the high and low values. The values for each parameter was selected, such that the higher (+) value was obtained by increasing from an initial setting until the presence of flash was notable. The lower value (-) was obtained by decreasing the parameter value until notable defects started to appear (for example, incomplete filling or low edge definition). The parameter values obtained are listed in table 3 and the processing run values in table 4. Parameters held constant were cooling time: 17 s, metering volume 210 mm^3 , injection speed 250 mm s^{-1} .

A Design of Experiments (DoE) method was used [21] for managing the results. The statistical model adopted for the study was the half fractional-factorial design. The term “fractional” indicates that a statistical significant percentage of all the possible tests was undertaken. With this approach, the full experimental test number (32, that is 2^5) were halved to 16 experimental tests (half) with a final resolution of V. Resolution V means that the design adopted will not exhibit alias between the main effects. The matrix of the statistical model it is reported in table 4. For this work a confidence limit of 95% was adopted.

2.5 Experimental design and procedure

The polymer used in the present study was a Polyoxymethylene (POM) BASF Ultraform® W2320 003 (melting point 166°C, tensile strength at room temperature 65MPa, linear thermal expansion coef. $0.6 \times 10^{-4} \text{ mm mm}^{-1} \text{ C}^{-1}$). The semi-crystalline POM was selected because it was expected to display different behaviour in parallel and normal directions [22, 23]. The equipment used for micro-moulding was a Battenfeld Microsystems 50.

Table 4 reports the combination of processing parameters investigated for determining the effect in terms of shrinkage. Each run was performed for an uninterrupted number of cycles, then five specimens were selected for shrinkage measurements.

2.6 Metrology Protocol

Several different methods are reported in the literature for measuring micro-dimensions, such as optical, laser beam based interferometry, volumetric, mechanical or rheometrical. However, there is sporadic information on accuracy. Because of the small specimen measurement dimensions and the specimen thickness, an optical method (TESA Visio 300), was chosen for its high accuracy ($\pm 1 \mu\text{m}$) and minimisation of mechanical stresses during measurement.

There is no specific protocol for the optical measurement of specimen dimensions in the cited standards. Therefore a protocol was devised and implemented. This is illustrated in figure 4. As the corner close to the gate was always present (figure 1) this was chosen as the zero point. The specimen was moved 5 mm to point 1. A line position was measured. The specimen was moved parallel to the flow direction until the opposite edge was reached. A second line position was measured (2). The specimen was moved back by 5 mm and then moved across the flow direction until the edge was found. A line position was measured (4). The specimen was moved in the opposite direction to the other edge. A final line position was measured (5). The same equipment was used to measure the mould cavity dimensions.

3. Results

3.1 Shrinkage measurements

Table 5 shows the effect of each processing parameters combination in terms of shrinkage, defined as reported in table 1.

3.2 Statistical analysis of moulding shrinkage in parallel to flow direction

The Pareto Chart depicted in figure 5 represents the mould shrinkage in parallel to the flow direction. It shows the magnitude and the effect of single and combined process parameters, along with a reference line indicating statistical significance. The processing parameters are labelled as A (hold time), B (hold pressure), C (injection pressure), D (mould temperature) and E (melt temperature). The combined influence of two of these parameters is described using two of the above letters.

The only statistically significant parameter was the mould temperature.

Figure 6 reports the Main Effect chart of mould shrinkage in the parallel to flow direction. Each Main Effect chart analyses the influence of a single factor, the slope of the line representing the magnitude and direction of the effect on the response. Vertical axes represent the shrinkage percentages reported in table 5. The mould temperature is the factor with highest magnitude; higher mould temperature led to a decrease in shrinkage.

3.3 Statistical analysis of moulding shrinkage in normal to flow direction

Figure 7 is the Pareto Chart of mould shrinkage in normal to flow direction. Again, the mould temperature was the only statistically significant factor. Figure 8 reports the Main Effect chart of mould shrinkage in normal to flow direction. The mould temperature is the factor with higher magnitude, but – opposite to the flow direction case - lower mould temperature led to a decrease in shrinkage.

3.4 Statistical analysis of post moulding shrinkage in parallel to flow direction

The Pareto Chart of post-moulding shrinkage in the parallel to flow direction did not show statistically significant effects.

3.5 Statistical analysis of post moulding shrinkage in the normal to flow direction

Figure 9 reports the Pareto Chart of post-moulding shrinkage in normal to flow direction. The combined effect of hold time and mould temperature has a statistically significant effect on shrinkage. The Main Effects plot is not reported because it is not used for describing the combined effect.

3.6 Statistical analysis of total shrinkage in the parallel to flow direction

Figure 10 is the Pareto Chart of total shrinkage in parallel to flow direction. Three factors had statistically significant effects on shrinkage: the mould temperature, hold pressure and melt temperature. In addition, two combinations of factors were statistically significant: hold pressure with mould temperature and mould temperature with melt temperature.

Figure 11, shows the corresponding Main Effects plot of the magnitude of effect. Increasing the value of either mould temperature, hold pressure or melt temperature led to a decrease of shrinkage.

3.7 Statistical analysis of total shrinkage in normal to flow direction

In contrast to the total shrinkage parallel to the flow direction, the total shrinkage normal to the flow did not show any statistically significant effects.

4. Discussion

Data obtained from the microscale mould could be used to detect statistically significant effects and to discriminate between the factors that affected the parallel and normal shrinkage. Further studies can take advantage of this approach for optimizing the processing parameters and comparing different feedstocks at the micro-scale.

Table 6 summarises the effects observed. Clear differences in shrinkage between parallel and normal to the flow direction were found. Furthermore, differences between moulding, post-moulding and total shrinkage were observed.

Mould temperature affected moulding shrinkage both parallel and normal to the flow but the direction of the effect was different. For post-moulding shrinkage, only shrinkage normal to the flow was affected by factors investigated in this study: a combination of holding time and mould temperature.

However for total shrinkage, only shrinkage parallel to the moulding direction was affected, and this was affected by three factors and two combinations of factors.

In the only prior micro-scale work [6], S_T values for semi-crystalline HDPE, are reported. Parallel to flow, mould temperature, melt temperature and hold pressure were significant. Normal to flow, in addition, the hold time was significant. For parallel to the flow, these results are the same as those presented here, except that the current paper also reports two combinations of factors that are significant. However, normal to the flow, the results are completely different, as in this work no significant effects were seen. The difference between the two sets of data is likely to lie in the standardisation of the mould used in this paper as a square design – the prior paper used a rectangular design.

There is no prior micro-scale work on S_M and S_P values that can be compared against this paper.

According to literature for macro-scale shrinkage, generally, increasing holding pressure decreases shrinkage. This particular effect was seen in the data presented here only for one case: total shrinkage parallel to the flow. At the macro-scale, the relationship between temperature factors and shrinkage is less well characterised. Fischer reports that for semi-crystalline polymers the shrinkage after an increase of mould temperature is a combinatorial effect with material parameters (percentage of crystallinity, internal stress), and processing parameters such as cooling rate. However, there is no reported differentiation between parallel and normal or moulding and post-moulding [15].

The complex connection between temperature factors and shrinkage at the macro-scale is confirmed by De Santis et al. [24] who analyzed the shrinkage trend of a semi-crystalline polymer in conventional injection moulding. The authors [22, 24] considered the densities of different phases and their degree of crystallinity, comparing the measured moulding shrinkage (at 10 minutes) with numerical modelling (though no data on statistical significance was reported). Moulding shrinkage was shown to reduce upon increasing the crystallinity. In a crystalline polymer, the temperature parameters (mould and melt temperature) are known to drive the crystal growth (rate of crystallization) by controlling the transition from melt to solid state [25].

5. Conclusions

This study investigated the feasibility of implementing international standards used to investigate shrinkage in conventional (macro) moulding at the micro-scale. The procedure proposed was able to discriminate between shrinkages in parallel and normal direction. The methodology presents a rigorous approach for detecting shrinkage in micro-scale injection moulding.

Five factors were investigated: the injection pressure, the holding pressure, the melt temperature, the mould temperature and the holding time. A DoE analysis was applied to determine the critical factors that affect the shrinkage in μ -IM. The critical factors identified were the mould temperature for the moulding shrinkage both parallel, and normal to, flow; the holding time-mould temperature combination for the post-moulding shrinkage in the normal direction; the mould temperature, the holding pressure, the mould temperature–holding pressure combination, the melt temperature and the melt temperature-mould temperature combination for the total shrinkage in the parallel to flow direction. No statistically significant effects were observed for total shrinkage normal to the flow, and post-mould shrinkage parallel to the flow.

Acknowledgements

The authors would like to thank Mr. J. Hedge and Dr. I. Walton for technical support during the mould manufacturing step.

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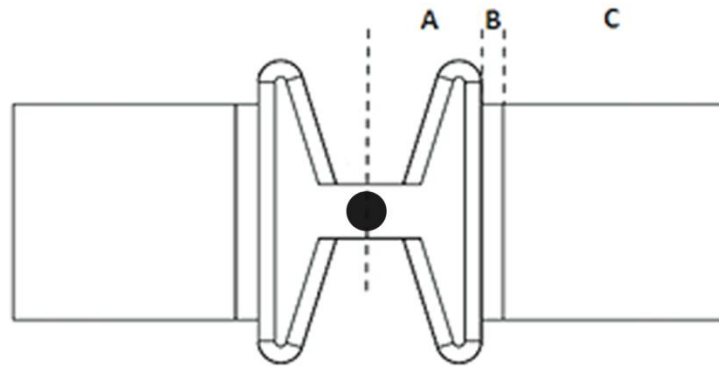


Figure 1. Different parts of mould: the runner (A), the rectangular gate (B), the square specimen (C). The black point represent the sprue.

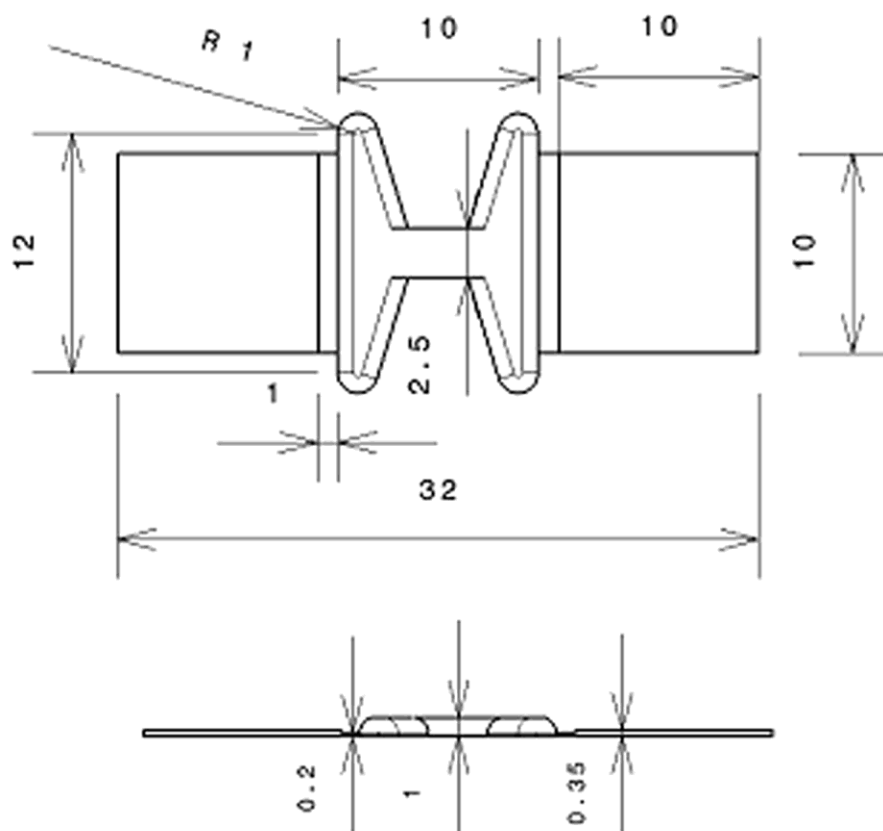


Figure 2. Dimensions of mould design in millimetres.

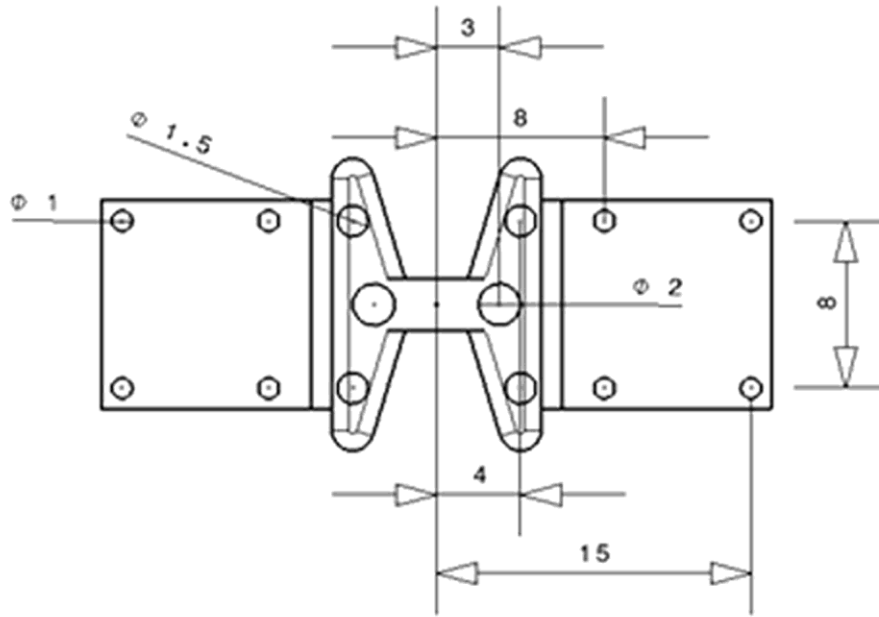


Figure 3. Micro-mould pin positions.

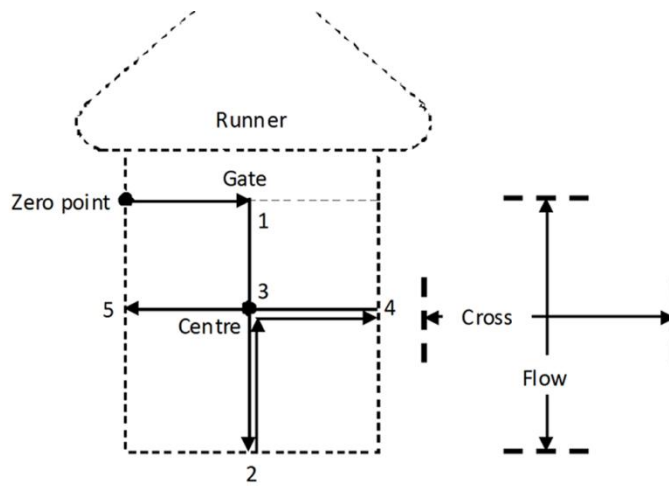


Figure 4. Measurement protocol adopted.

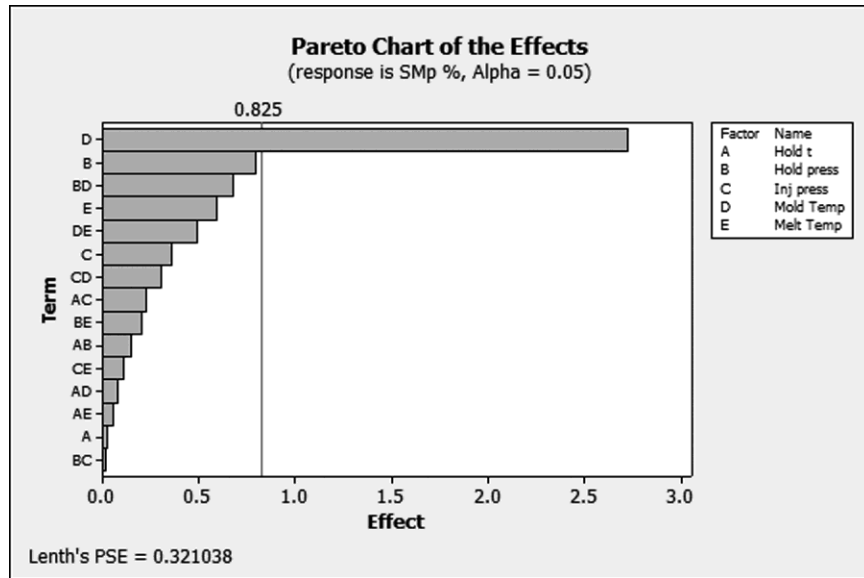


Figure 5. Pareto Chart of POM moulding shrinkage in parallel direction.

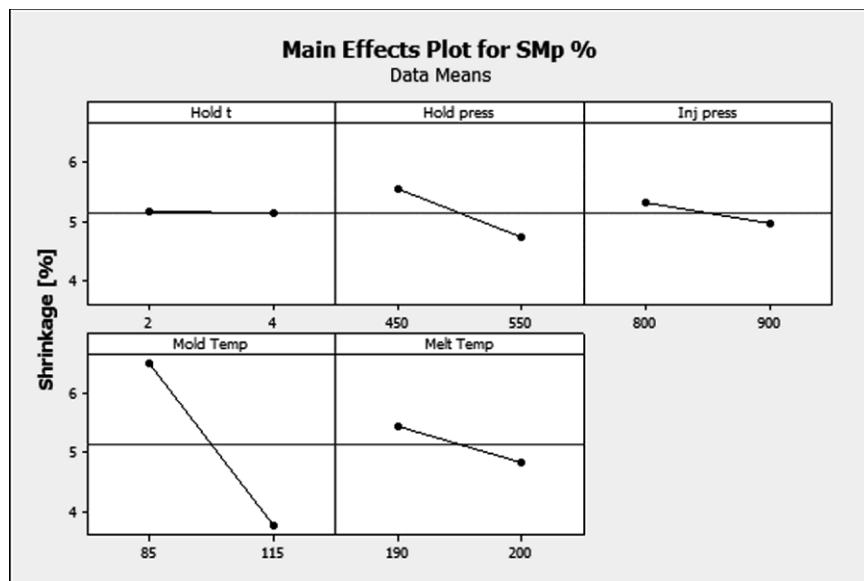


Figure 6. Main Effects of POM moulding shrinkage in parallel direction.

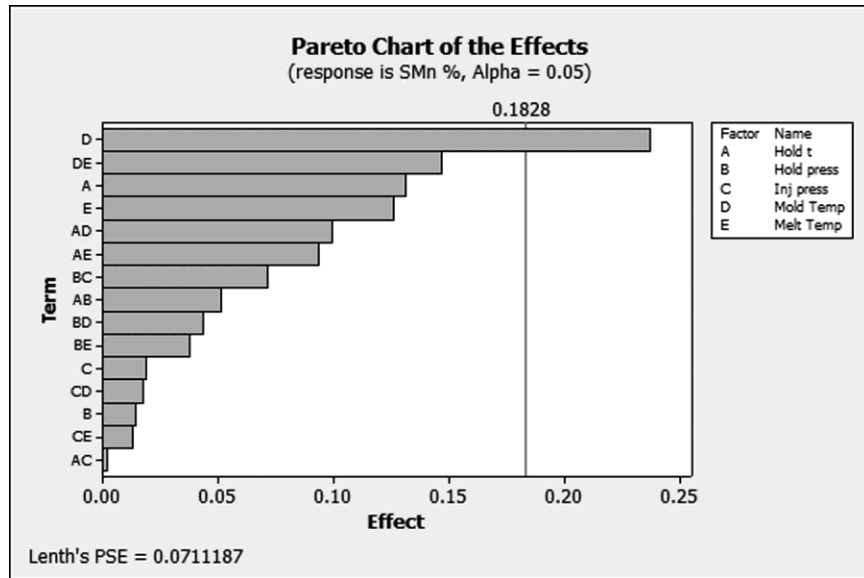


Figure 7. Pareto Chart of POM moulding shrinkage in normal direction.

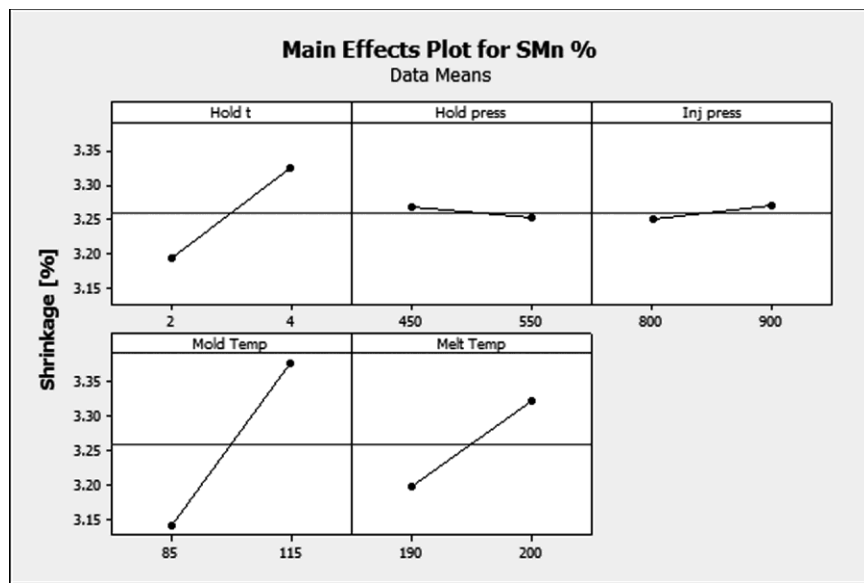


Figure 8. Main Effects of POM moulding shrinkage in normal direction.

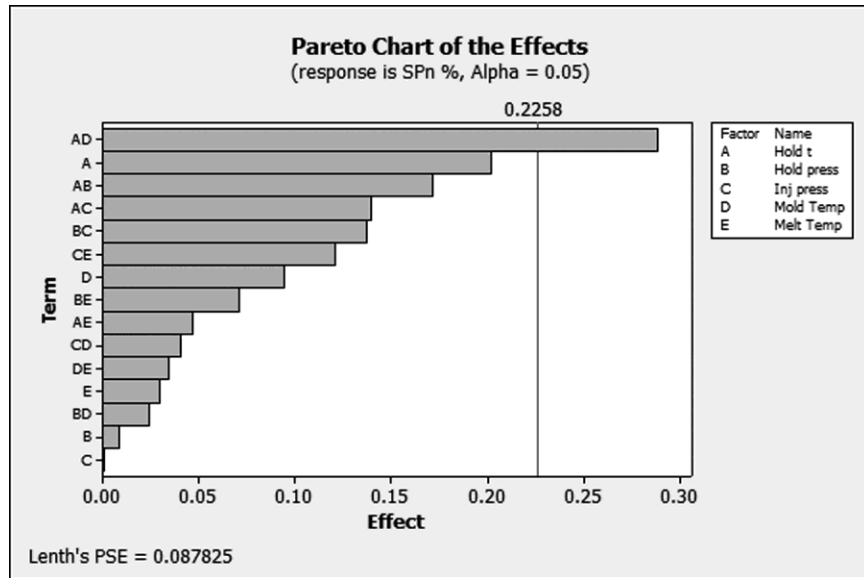


Figure 9. Pareto Chart of POM post-moulding shrinkage in normal direction.

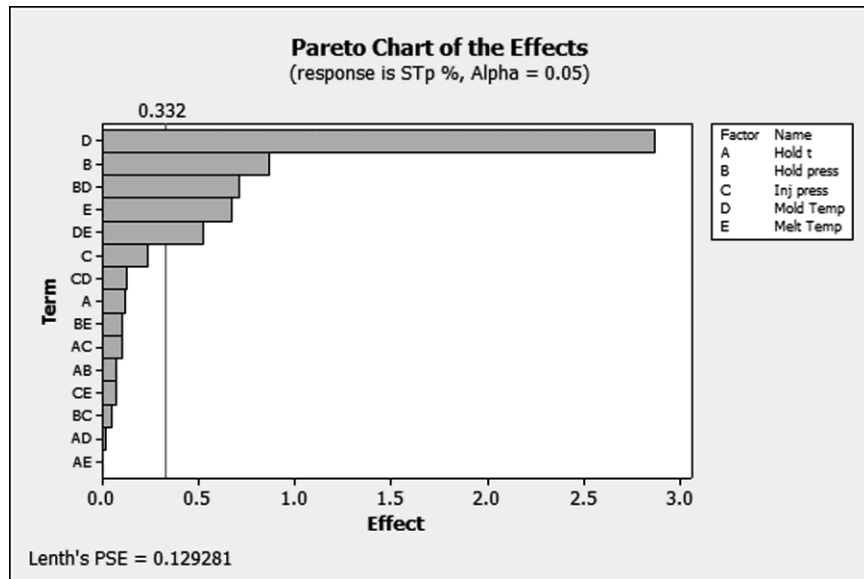


Figure 10. Pareto Chart of total shrinkage in parallel direction.

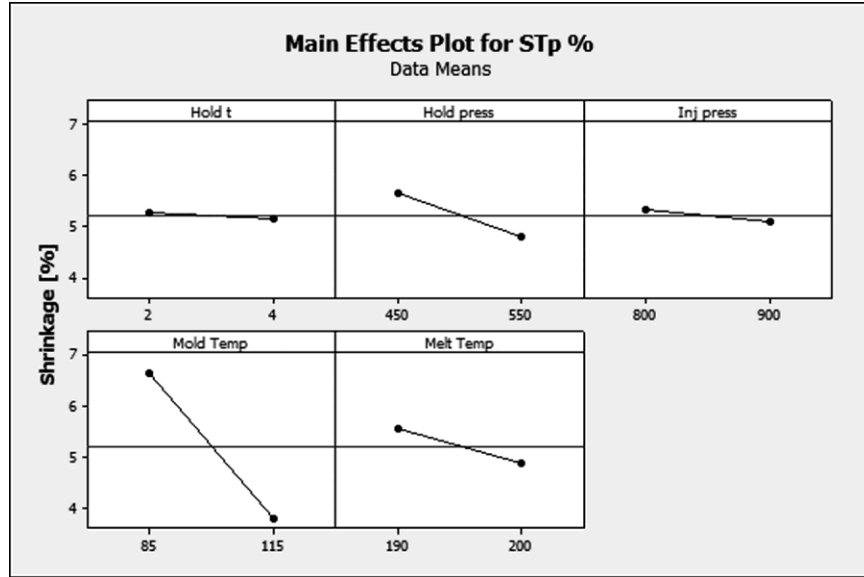


Figure 11. Main Effects of total shrinkage in parallel direction.

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Moulding Shrinkage	$S_{Mp} = 100 \frac{l_c - l_1}{l_c}$; $S_{Mn} = 100 \frac{b_c - b_1}{b_c}$
Post-moulding Shrinkage	$S_{Pp} = 100 \frac{l_1 - l_2}{l_1}$; $S_{Pn} = 100 \frac{b_1 - b_2}{b_1}$
Total Shrinkage	$S_{Tp} = 100 \frac{l_c - l_2}{l_c}$; $S_{Tn} = 100 \frac{b_c - b_2}{b_c}$

Table 1. Mathematical formulas for measuring the shrinkage. Subscripts p and n are respectively parallel and normal measurement to flow direction; c is related to the centre of mould cavity; 1 and 2 are referred to 1 hour and 24 hour measurements, l is the length and b is the breadth.

Processing Parameters	Expected effect from Fischer [15] with increase in magnitude of processing parameters	
Injection pressure	Decreases (usually)	↓
Holding pressure	Decreases	↓
Melt temperature	May be either	—
Mould temperature	Increases	↑
Holding time	Decreases	↑

Table 2. Effect of selected parameters in terms of shrinkage according to Fischer.

Process Parameters	Initial Values	Value +	Value -
Injection pressure [bar]	850	900	800
Holding pressure [bar]	500	550	450
Melt temperature [°C]	195	200	190
Mould temperature [°C]	100	115	85
Holding time [s]	3	4	2

Table 3. The values of processing parameters tested.

Processing parameter combinations						Half Fractional Factorial matrix				
Run	Hold time [s]	Hold press [bar]	Inj. press [bar]	Mould temp [°C]	Melt temp [°C]	Hold time	Hold press	Inj. press	Mould temp	Melt temp
1	2	450	900	85	190	+	-	+	-	+
2	4	450	900	85	200	+	-	-	+	+
3	4	550	800	85	200	+	+	-	-	+
4	4	550	900	85	190	+	-	-	-	-
5	2	550	800	115	190	+	+	+	-	-
6	2	550	900	85	200	-	-	+	-	-
7	2	550	800	85	190	+	-	+	+	-
8	4	450	900	115	190	-	+	+	-	+
9	2	450	800	85	200	-	+	-	+	+
10	4	450	800	85	190	-	-	+	+	+
11	2	450	900	115	200	+	+	-	+	-
12	4	550	800	115	190	+	+	+	+	+
13	2	550	900	115	190	-	+	-	-	-
14	4	450	800	115	200	-	+	+	+	-
15	4	550	900	115	200	-	-	-	-	+
16	2	550	800	115	200	-	+	-	+	-

Table 4. Matrix of half fractional factorial design and processing values.

Run	S_{Mp} [%]	S_{Mn} [%]	S_{Pp} [%]	S_{Pn} [%]	S_{Tp} [%]	S_{Tn} [%]
1	6.910±0.014	2.731±0.022	0.793±0.022	0.068±0.031	7.649±0.008	2.797±0.007
2	6.566±0.002	3.335±0.008	0.104±0.004	-0.440±0.012	6.664±0.001	2.909±0.001
3	5.067±0.005	3.227±0.008	0.043±0.008	-0.349±0.011	5.108±0.004	2.890±0.001
4	5.977±0.008	2.862±0.003	0.122±0.015	-0.061±0.004	6.092±0.009	2.802±0.002
5	3.809±0.002	3.178±0.001	0.081±0.004	0.040±0.001	3.887±0.002	3.217±0.001
6	4.676±0.010	2.932±0.004	0.310±0.016	-0.010±0.006	4.972±0.009	2.923±0.001
7	6.783±0.010	2.825±0.001	-0.009±0.015	-0.005±0.002	6.775±0.007	2.820±0.001
8	3.722±0.001	3.228±0.002	0.007±0.002	0.026±0.002	3.728±0.001	3.253±0.001
9	6.989±0.004	2.838±0.001	-0.022±0.007	0.566±0.009	6.968±0.004	3.388±0.009
10	7.948±0.010	2.824±0.001	0.007±0.017	-0.489±0.008	7.954±0.010	2.350±0.008
11	3.660±0.001	3.177±0.001	0.010±0.001	-0.083±0.002	3.670±0.001	3.096±0.001
12	3.600±0.001	3.170±0.001	0.086±0.001	-0.016±0.001	3.683±0.001	3.155±0.001
13	3.640±0.001	3.199±0.001	-0.011±0.001	-0.023±0.001	3.629±0.001	3.177±0.001
14	3.616±0.001	3.268±0.001	0.024±0.001	0.030±0.002	3.640±0.001	3.297±0.002
15	3.437±0.001	3.134±0.001	-0.065±0.002	0.178±0.003	3.373±0.001	3.306±0.004
16	3.662±0.002	3.116±0.001	-0.029±0.004	-0.089±0.001	3.633±0.002	3.030±0.001

Table 5. Shrinkage results (the shrinkage nomenclature was defined in table 1).

S_{Mp}	Mould temperature	↓
S_{Mn}	Mould temperature	↑
S_{Pn}	Holding time & mould temperature	—
S_{Tp}	Mould temperature	↓
	Holding pressure	↓
	Mould temperature & holding pressure	—
	Melt temperature	↓
	Melt temperature & mould temperature	—

Table 6. DoE results of the effect of processing factors. The arrows indicate - for single processing parameters - whether a factor increasing causes an increase (↑) or decrease (↓) in shrinkage.

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2013-06-01T00:00:00Z

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Daniele Annicchiarico, Usama M. Attia and Jeffrey R. Alcock, A methodology for shrinkage measurement in micro-injection moulding, Polymer Testing, Volume 32, Issue 4, June 2013, Pages 769–777.

<http://dx.doi.org/10.1016/j.polymertesting.2013.03.021>

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