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Analysis of the influence of part thickness on the replication of micro-structured surfaces by injection molding

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ABSTRACT

The achievement of an adequate accuracy of the microinjection molding process applied to the replication of micro-features is a complex task. The selection of process parameters and the geometry of the molded part influences the filling flow of the melt inside the mold cavity as well as the final quality of the replicated micro-features. In this work, the replication of a micro-structured surface is studied in relation to the thickness of the molded part. In particular, the effects of a rapid heat cycle molding process, cavity air evacuation, part thickness are experimentally evaluated. The analysis of the experimental data showed that the thickness of the main flow region and the molding temperature are significant factor affecting the replication quality. In particular, a combination of small cavity thickness and a high mold temperature (above T_g) can be effective in increasing the replicated height of micro-features. Experimental results also point out the correlation between the holding pressure and the distance from the injection location.

An effective approach to the design of both the part and the process, should consider that the selection of process parameters must be related to design of the mold cavity.

1. Introduction

Microinjection molding is the process of replicating micron or even sub-micron features from metallic molds to polymeric products, which can have overall dimensions in the macro-range and areas with micro-features. The accuracy of this technology greatly depends on features size, aspect ratio and surface area. In particular the aspect ratio achievable in replicating micro-features is one of the most important capability of a micro fabrication technology and determines the manufacturing constraints of a given process/material combination [1].

A typical injection molded part with micro-features can be described as a substrate, usually called the main flow region, having micro-features located on part of its surface [2]. The substrate has the function of supporting the microstructures, and its geometry affects the filling flow pattern of the polymer melt and consequently the replication quality of the molded part. The understanding of the characteristics of the flow in thin-wall cavities, typical of microinjection-molded parts, is essential in order to properly design both the part and the process.

The filling of the substrate is characterized by an essentially flat advancing flow front. On the other hand, the filling of micro-features is more complex. The molten polymer initially flows into the substrate, where it encounters lower resistance to the flow, and hesitates at the entrance of micro-features until the cavity pressure is high enough to win the flow resistance in the micro-feature, which usually occurs at the end of the filling [3]. According to this phenomenon, commonly known as the 'hesitation effect', the packing pressure is regarded as the main driving force that compels the polymer into the micro-features [4]. However, this behavior could result in incomplete replication, because the filling time of the substrate is usually greater than the freezing time of the injected material at the entrance of the micro-features. The solidified skin formed on the surface of the main cavity opposes to the filling reducing the effect of the packing pressure [5]. Indeed, the packing pressure could slightly improve the replication of micro-features, however its main effect is to counteract the part shrinkage and therefore to maintain the replication achieved during the filling stage [6].

The key factor determining the filling length of micro-features is the growth of the pressure at their entrance, which depends on polymer properties, process settings and geometric parameters [7]. High values of process parameters, such as mold temperature and injection speed can improve the flow in the cavity [8]; however, a complete replication of high aspect ratio micro-features is challenging even using state-of-the-art high-speed injection systems, rapid heat cycle molding and vacuum mold venting [9].

The geometry of the molded part can influence the melt filling by directly affecting the pressure profile in the macro cavity. In particular, the thickness of the substrate, which is much larger compared to the micro-features, affects the replication quality by changing the evolution of the cavity pressure generated during the injection phase [10]. Moreover, substrate thickness is critical because it determines the cavity filling time for a given injection speed [11].

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The influence of cavity thickness on replication quality is not consistent among different researches reported in the literature. Some authors indicated that the filling capability of the melt declines rapidly with the reduction of part thickness [12]. Due to rheological reasons, it was suggested that injection molded parts with a sufficient replication quality need a minimum thickness of approximately 2 mm [13]. Conversely, other reported that a decrease of part thickness could enhance the filling of micro-features by counteracting the rapid cooling [14]. Thus, a careful consideration of part geometry is required to control the quality of the molded parts [15]. In particular, the value of the thickness of the main flow region should take into account both aspects of the problem. The relation between the replicated height of micro-features and the thickness of the main flow region needs to be further investigated, especially in combination with state-of-the-art microinjection molding technologies, such as rapid heat cycle molding (RHCM) and cavity air evacuation.

In this work, the influence of cavity thickness on replication quality was studied analyzing the replication of micro-pillars distributed over a large surface. The evacuation of air from the cavity and RHCM were used in order to enhance the degree of replication in combination with the geometry of the cavity.

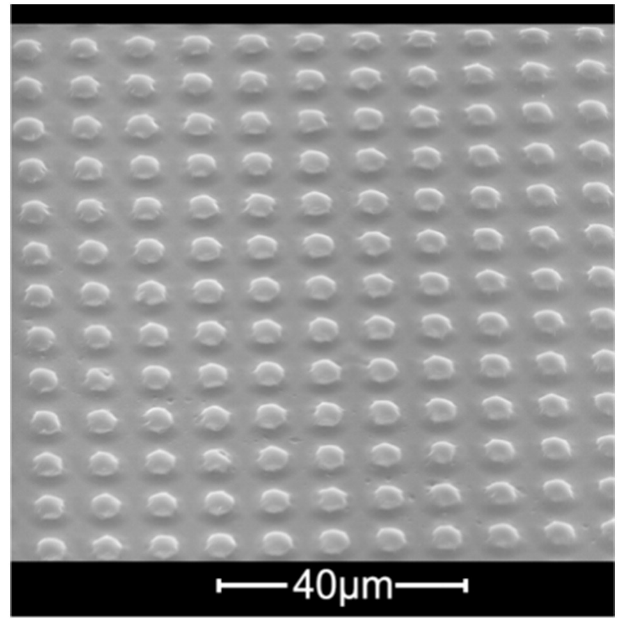

Fig. 2. SEM photo of the replicated polymeric surface.

2. Experimental

2.1. Part and mold design

The molded part is a cylindrical support having a diameter of 5.9 mm (Fig. 1) and a micro-pillared surface topography (Fig. 2). The micro-pillars are characterized by a diameter of 4 μm and an interspace of 10 μm, as shown in Fig. 3. In the mold, the cylindrical cavity was positioned on the moving plate, at the end of a rectangular cold runner (20 mm long, 2 mm wide and 1.5 mm thick), as shown in Fig. 1. The cavity was machined on the mold by the means of a 5 axis micro-milling machine (Kugler Micromaster 5×), that allowed the achievement of a good finishing. Two different versions of the cavity were used, with thicknesses of 1 and 2 mm, respectively.

The mold insert with micro-features was manufactured from a rectangular piece of 39NiCrMo3 steel, according to the following operations: lapping, machining, photolithography. The steel insert was initially sandpapered (grit: 180, 600, 1200) and then refined with abrasive particles (6, 3, 1 μm) in order to achieve a surface with Ra < 0.05 μm for the subsequent deposition of the masking film. The lapped insert was then machined to obtain the desired shape to mount it on the fixed plate of the mold.

In a subsequent phase, a TMSPM-Zr hybrid sol–gel resist was spin coated at 300 rpm for 30 s over the polished surface of the insert. The desired pattern of micro-pillars was then realized by exposing the insert to a 4 J/cm^2 ultraviolet light for 10 s through an appropriate mask. After the photolithography process, the resist was developed for 70 s in an acidic solution (ethyl alcohol:dimethyl ketone 100:1 solution) to remove the resist in the areas where it was not cross-linked by the UV source. The insert was then hard-baked at 100 °C for 1 h.

The mold insert that was then characterized using an Atomic Force Microscope (DME DS 95-200) having a nominal scanning range of 200 × 200 μm^2. The insert was measured in three areas along the diameter of the cavity (close to the gate, in the center and at the opposite side from the gate). The average holes depth is 5.000 ± 0.094 μm. Hence, the micro-holes are characterized by an aspect ratio of 1.25.

2.2. Material

A commercial polystyrene (Total PS Crystal 1540) was used in the experiments. This material is suitable for microinjection molding applications, due to its high flowability, good biocompatibility, high optical clarity, high transparency and high impact strength. Table 1 reports the main properties of the polymeric material.

2.2.1. Manufacturing system

Injection molding experiments were carried out using a Wittmann-Battenfeld MicroPower 15 state-of-the-art μIM machine (maximum injection speed: 750 mm/s, maximum clamping force: 150 kN). The machine has an injection system divided into a 14 mm plasticizing screw and a 5 mm injection plunger.

In order to realize a rapid heat molding cycle, eight electrical heating cartridges were installed into the mold, while the cooling was realized with cold water circulating in the mold at 5 °C. The variotherm system control was realized with an external control unit. The mold was heated up to a set temperature prior to cycle start. At the end of the injection phase, the heating system was switched off and the cooling water was pumped into the mold channels.

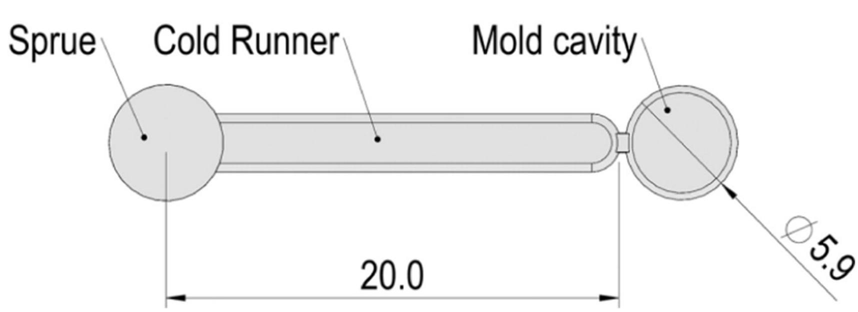

Fig. 1. Geometry of the molded part.

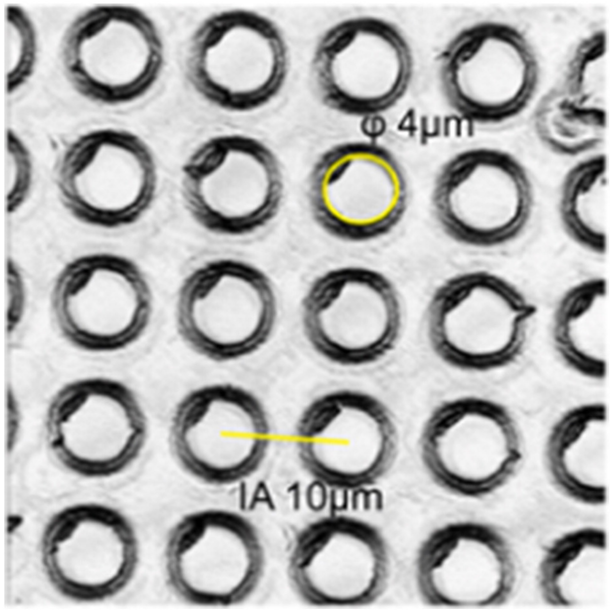

Fig. 3. Micro-pillars scanned with the optical profiler.

In addition, a vacuum pump was connected to the mold to evacuate the air from the cavity before the injection phase. An O-ring was positioned on the moving plate of the mold, to surround the cavity and seal the mold-parting plane. The pump was connected to the mold through a vent that was machined at the opposite side of the gate and inside the sealed area. The cavity pressure before the injection was set at 6 mbar. The vacuum venting was applied for 10 s before the injection and for all its duration [16].

2.2.2. Characterization of the molded parts

The replication accuracy of the polymeric micro-features has been evaluated with a state-of-the-art 3D optical profiler (Sensofar PLu neox), used in confocal mode with 20× and 100× objectives. Its main characteristics are reported in Table 2. Vertical calibration of the instrument was performed using silicon depth standard type A1 (ISO 5436-1). The optical profiler allowed the reconstruction of surface topography, and so the quantitative measurement of the replicated micro-pillars height. The use of this measurement technology did not permit the characterization of the shape of the pillars [17], as the maximum measurable slope is of 51°.

The measurements were carried out in three positions along the diameter of the molded part, in an area of 127.32 × 95.45 μm^2 (Fig. 4).

3. Experimental investigation

The investigation was designed according to a two-level, five-factor, 1/4 fractional plan [18]. This fractional design is of resolution III, hence providing sufficient information about the main effect of the factors. However, two-factor interactions are aliased. This plan allowed for a relatively reduced number of experiments to be carried out with an acceptable compromise in terms of results accuracy, only regarding the effects of main factors. For each run, the parts produced in the first 10 cycles were discarded in order to stabilize the process, and then the following part was collected for the metrological characterization. Each run was repeated three times in a completely randomized order for a total of 24 produced samples. In order to minimize interference from external variability sources, the polymer was taken from a single batch.

Table 1
Main properties of the total PS crystal 1540.

Property	Units	Test method	PS
Density	g/cm^3	ISO 1183	1.00
MFI (200 °C–5 kg)	g/10 min	ISO 1133	12.0
T_g (10 °C/min)	°C	ISO 1157	100

Table 2
Main characteristics of the 3D optical profiler.

	20×	100×
Numerical aperture	0.45	0.90
Max. slope (deg.)	21	51
Field of view (μm)	636 × 477	127 × 95
Spatial sampling (μm)	0.83	0.17
Optical res. (μm)	0.31	0.15
Vertical res. (μm)	<20	<2

3.1. Investigated factors

The analyzed factors were the mold temperature (T_m), the injection speed (V_{inj}), the holding pressure (P_{hold}), the thickness of the substrate (t) and the presence of cavity air evacuation (E_a).

During the molding experiments, the following parameters were fixed to a constant value suggested by the literature and technological considerations:

- barrel temperature: 240 °C
- holding pressure time: 7 s
- cooling time: 12 s
- vacuum valve:
  - on, with time monitoring:
  - after build up clamping force;
  - air evacuation time before the injection: 10 s;
  - air evacuation time after the start of the injection: 7 s;
  - in cavity pressure after air evacuation: 6 mbar.

The parameters selected for the experimental plan were considered to be the ones affecting the capabilities of the process in terms of replication quality, as resulting from a literature review, recommendations of the material supplier and technological limits of the available experimental setup. The levels for the investigated parameters are reported in Table 3.

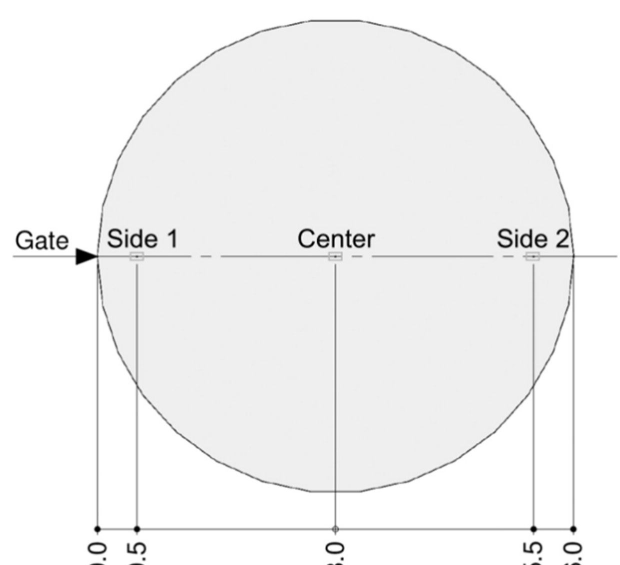

Fig. 4. Measured areas for the characterization of the height of micro-pillars; all quotes are expressed in mm.

Table 3
Process parameter setting and other factors for the factorial plan.

Level	T_m (°C)	V_{inj} (mm/s)	P_{hold} (bar)	t (mm)	E_a
(−1)	80	450	250	1	Off
(+1)	120	750	500	2	On

3.2. Response variable

The replicated height of the pillars was chosen as response variable for the statistical analysis of the experimental data. For each treatment, all replications were measured in three areas (Side1, Center, Side2) along the diameter of the molded part, as shown in Fig. 4.

Fig. 5 shows the topography of the surface of a molded part in the scanned area. In each scanned area, the profiles of five micro-pillars were considered, for a total of fifteen micro-pillars analyzed for each replication. Therefore, the value attributed to each measurement corresponds to the height of a characteristic micro-pillar that is an average value of five pillars considered within the scanned area. The value assigned to each replication is the average of the measurements in the three scanned areas.

3.3. Analysis of the factorial plan

Table 4 reports the average results of the measurements in the three positions (Side1, Center, Side2) for all the combinations of the designed factorial plan. For each treatment the 1/4 fraction factorial design has been analyzed in order to evaluate which factors and interactions are significant in determining the quality of the replication. A General Linear Model was used to perform a univariate analysis of variance (ANOVA) for the designed factorial plan. The terms included in the model are all the main factors implemented in the design of the experimental plan. In order to evaluate the statistical significance of the factors included in the model the ANOVA table was considered. In particular, the following parameters were calculated:

- Adjusted sums of squares: measurements of variations for different components of the model;
- Adjusted mean squares: indications of how much variation a term or a model explains, assuming that all other terms are in the model, regardless of the order they were entered;
- *F*-value: it is the statistic test used to determine whether the term is associated with the response;
- *P*-value. It is a probability that measures the evidence against the null hypothesis. Lower probabilities provide stronger evidence against the null hypothesis.

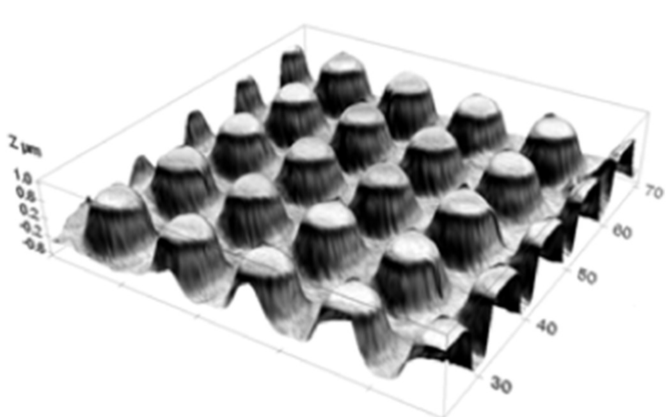

Fig. 5. 3D topography of the PS replication acquired with the optical profiler.

Table 4
Results of the factorial plan.

Level	T_m [°C]	V_{inj} [mm/s]	P_{hold} [bar]	E_a	t [mm]	Side1	Center	Side2	H_{avg} [µm]	Std. Dev. [µm]
A	80	750	500	Off	1	1.67	1.26	1.34	1.42	0.22
B	80	750	250	Off	2	0.74	0.83	1.33	0.97	0.32
C	80	450	500	On	1	1.64	1.37	1.19	1.40	0.23
D	80	450	250	On	2	0.74	0.92	1.63	1.10	0.47
E	120	450	250	Off	1	4.17	4.16	4.53	4.29	0.21
F	120	450	500	Off	2	3.56	2.99	3.08	3.21	0.31
G	120	750	250	On	1	3.56	3.75	4.20	3.84	0.33
H	120	750	500	On	2	4.07	3.62	3.06	3.58	0.50

The *P*-value is the parameter that was used for the statistical identification of significant parameters, the threshold value was fixed at 0.05. In other words, factors having a *P*-value inferior to 0.05 can be considered statistically significant to the selected response.

The considered plan is characterized by resolution III, thus it enables the statistical estimation of main effects, but the effects of two-factor interactions cannot be statistically evaluated. Thus, the following discussions will only consider the effects on the replication of main factors.

4. Results and discussion

4.1. Factors affecting replication quality

The results of the ANOVA, reported in Table 5, indicate that only the thickness of the substrate and the mold temperature significantly affect the replicated height of the micro-pillars. As shown in Fig. 6(a), increasing the thickness of the substrate from 1 to 2 mm yields a reduction of the average height of 19%. In fact, a higher thickness tends to limit the replication during the injection phase, due to a lower rising of cavity pressure. Indeed, the growth rate of cavity pressure is related to the pressure drop in the cavity, which is greater with a smaller thickness. Moreover, the main effect of the holding pressure resulted not significant, restraining the possibilities for the increase of the filling ratio in the packing stage [5,6]. Conversely, a smaller thickness favors the increase of the replicated height of micro-pillars by increasing the in-cavity pressure at the entrance of micro-cavities during the injection phase. Additionally, a thinner cavity favors a rapid filling allowing the polymer the time to fill micro-features before solidification starts. In fact, the replication depends on how quickly the polymeric melt flows into the micro-cavities before solidifying.

The mold temperature is also a significant parameter. As displayed in Fig. 6(b) an increase of mold temperature from 80 to 120 °C lead to an increase in the average height of micro-pillars from 1.2 to 3.7 µm, that is more than 300%. Mold temperature is important because of its influence on the thermal gradient between the cavity surface and the injected polymer. The viscosity of the polymer melt is highly dependent on temperature, thus, it is crucial to keep the material flowing at high temperature during the filling phase. This was achieved by maintaining the highest possible value of the barrel temperature according to the materials datasheet and by adopting a rapid heat molding cycle.

Table 5
Anova table for the designed experiment.

Factor	Adj SS	Adj MS	F	P
T_m	37,765,903	37,765,903	901.84	0.000
V_{inj}	12,531	12,531	0.30	0.591
P_{hold}	123,710	123,710	2.95	0.103
E_a	401	401	0.01	0.923
t	1,631,238	1,631,238	38.95	0.000
Error	753,776	41,876		

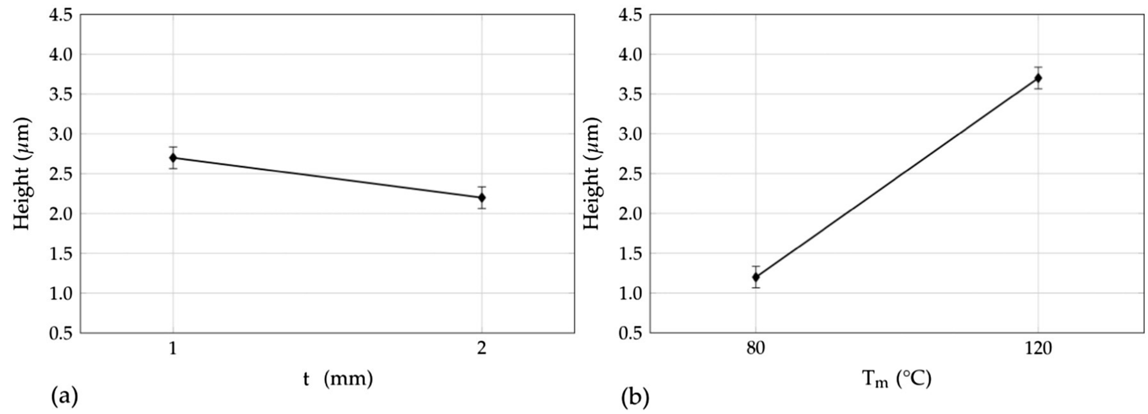

Fig. 6. Main effect plot for the thickness (a) and the mold temperature (b).

4.2. Influence of the distance from the injection location

The experimental results (Table 4) indicated a possible relation between the replicated height of the micro-features and their distance from the injection location.

Fig. 7 displays the difference δ from the replicated height in the Side1 position and the height in the Side2 position:

$$\partial = H_{Side1} - H_{Side2}$$

Positive values of δ indicate that the replication decreases moving from the injection location to the end of the cavity, while negative values of δ stand for the opposite trend of replication.

Considering the influence of process parameters on δ, it can be argued that high values of the holding pressure produce a higher replication degree closer to the injection location. The pressure is higher in this region, but quickly reduces going far from the gate because of the frictional shear forces caused by the resistance to flow. The same replication trend applies when molding with both lower and higher values of the mold temperature. Indeed attributing more importance to the contribution of the holding stage to the replication of the micro-features located closer to the gate.

Comparing treatments A and C it could also be observed that the injection speed especially affects the replication of the features located far from the gate (Side2). In particular, varying the injection speed from 450 to 750 mm/s produces an increase of 0.15 μm between the two calculated heights in Side2. This means that the pressure at the end of the filling is significant in determining the final replication of features located far from the gate.

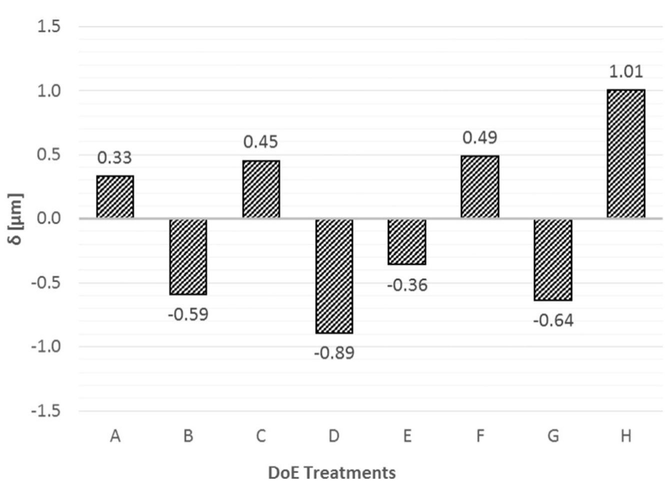

Fig. 7. Column chart for the difference between the replicated height in position Side1 and in position Side2.

5. Conclusions

The main goal of the current study was to determine the influence of cavity thickness on the replicated height of micro-pillars. In particular, its significance has been studied in relation with state-of-the-art technologies (rapid heat molding cycle and vacuum venting) that are widely considered effective in enhancing replication quality.

The analysis of the experimental data showed that the thickness of the main flow region is a significant factor affecting the quality of the replication. In particular, a smaller thickness favors the replication (+19%) by ensuring a quicker filling of the substrate, thus allowing time for the filling the micro-cavities before solidification starts. Furthermore, a smaller cavity allows a markedly higher growth of cavity pressure during the injection phase that enhances the filling of micro-cavities.

The molding temperature resulted also significant, indicating that the average replication of the micro-features can be maximized by adopting high molding (above T_g) and a small cavity thickness.

Considering the replication in relation with the distance from the injection location, it was observed that the holding pressure can be effectively exploited to improve the replication close to the gate, where the pressure is higher. The effect of the injection speed resulted significant in determining the replicated height of micro—features at the end of the cavity.

Nomenclature

T_m	mold temperature
V_{inj}	injection speed
P_{hold}	holding pressure
E_a	cavity air evacuation
t	substrate thickness
T_g	glass transition temperature
Adj SS	adjusted sums of squares
Adj MS	adjusted mean squares
F	*F*-value
P	*P*-value
δ	difference between replication in positions Side1 and Side2
H_{avg}	average replicated height

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