A study of hot embossed microchannels using confocal microscopy

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ABSTRACT

Micro molding and hot embossing are among the most popular techniques for fabricating microstructures in polymeric materials, due to the relatively low cost of replication. In this paper we present an investigation of a hot embossing process including the optimisation of operating parameters for polystyrene (PS) and polycarbonate (PC) substrate materials. Nickel mold inserts were made via excimer-laser ablation and Ni electrodeposition. Replication of structures, such as channels with different aspect ratios, were investigated and experimental results are discussed.

Keywords: Replication, Hot Embossing, Confocal microscopy.

1. INTRODUCTION

At the present time injection molding and hot embossing are among the most commonly used techniques for replication of polymer microdevices. Injection molding is the most popular method of replication for cost effective large-scale production, however, for the highest quality of microparts hot embossing is usually employed. Other advantages of the hot embossing process are small thermal cycle, and small thermal stresses [1-3].

The hot embossing process can be described as follows: The polymer substrate and master-mold are heated separately under vacuum to an equal and uniform temperature higher than glass transition temperature (Tg) of the polymer material. The master-mold is then pressed against the polymer substrate by a precisely controlled force. After a certain time the substrate and the mold are cooled to a temperature below the Tg while still applying the embossing force. The subsequent step of the process is deembossing, where the master-mold is separated from the substrate.

During recent years the hot embossing process has been thoroughly studied with a variety of micro and nano structures fabricated by hot embossing were presented [1-5]. The influence of substrates rheological properties [6] and surface tension effect during the hot embossing process [7] were investigated. From this published work it is apparent that the optimal temperature for embossing is fairly consistent (i.e. slightly above the materials Tg), but each system requires optimisation of the embossing time and pressure in order to achieve the desired structures.

Different techniques can be employed to fabricate the master mold for hot embossing which mainly depend on the size and geometry of the microstructures. From CNC machining of stainless steel for microstructures with dimensions in hundred micrometer range [3] to LIGA technologies for microstructures with dimensions in few micrometers range and high aspect ratios [8-10]. Nickel is among most commonly used materials for mold insert microfabrication [2], although Silicon has been proven to be a very good material for master mold fabrication [9]. Both of these materials are used due to a combination of their ease of fabrication and superior mechanical properties.

Two different approaches were used for optimisation of operating parameters of hot embossing process. For PS substrate material a method of Design of Experiments (DOE) was employed, which is widely used in conventional process optimisation [11-13]. It allows for a significantly reduced number of experiments in optimisation of process parameters in such processes as extrusion, injection molding, etc. As a response function, the fraction of error of cross-sectional area of embossed microstructures was chosen. The influence of embossing pressure, cycle time and embossing temperature on the quality of replication is presented in the form of second order equations. For PC substrate material, a conventional experimental approach was used, where two variables were kept constant during

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the sets of experiments and one was changed. For analysing results of hot embossing processes for PS and PC cross-sectional measurements of master mold and hot embossed microchannels made by confocal microscope were overlayed.

Scanning laser confocal microscope was particularly useful as it facilitates cross section measurements of microstructures without special preparation of the samples, such as cutting or sputtering, as is the case when using a scanning electron microscope (SEM).

2. EXPERIMENTAL

2.1 Mold insert manufacturing

Manufacturing process of master mold included excimer laser ablation of polycarbonate and subsequent nickel electro deposition. An excimer laser micromachining system (Exitech Ltd, S8000) was used to produce polymer molds. This system works by illuminating a chrome-on-quartz mask, and the image is demagnified by x10 to ablate the workpiece [14]. As the result of UV-laser ablation, two different polycarbonate molds were machined, as detailed in Table 1.

Master embossing shims were produced by electroforming into the molds using a Ni-sulphamate solution. As a result of these two processes Ni master molds were obtained with dimensions 25mm x 75mm and 300µm thickness. Microstructures on the master mold included structures which can be described as inverse channels with variable height and width. A single line mold was used in hot embossing of PS, and a double line mold was used in hot embossing process of PC.

An average aspect ratio $R_{av}$ was calculated using following equation:

$$R_{av} = \frac{2 \cdot H}{W_{bottom} + W_{top}}$$

<table>
<thead>
<tr>
<th>Line</th>
<th>Single line mold</th>
<th>Double line mold</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Line 1</td>
</tr>
<tr>
<td>Height [µm]</td>
<td>200</td>
<td>14</td>
</tr>
<tr>
<td>Width bottom [µm]</td>
<td>173</td>
<td>28</td>
</tr>
<tr>
<td>Width top [µm]</td>
<td>35</td>
<td>25</td>
</tr>
<tr>
<td>Average aspect ratio ($R_{av}$)</td>
<td>1.92</td>
<td>0.53</td>
</tr>
</tbody>
</table>

Table 1. Dimensions of microstructures patterned into mold inserts.

The single line mold used for hot embossing of PS and the double line mold used for hot embossing of PC.

2.2 Embossing equipment

A home made embossing tool consists of top and bottom stages, as shown in Figure 1 was used for all embossing experiments. The top stage can slide in vertical direction on four guiding posts, while the bottom stage is fixed. The top and bottom parts of the embossing tool have three 400-watt heating elements in each of them. Cooling is achieved by circulating compressed air. Also, there is a vacuum pump connected to the top part of the embossing tool, in order to remove air bubbles and vapour during the embossing process.
2.3 Hot Embossing Process

An isothermal hot embossing process was performed, whereby the master mold and substrate material were heated separately to the same temperature before the actual embossing step is performed [15]. The process can be described as follows: the master mold and polymer substrate are placed into the embossing chamber. The chamber is closed by lowering the upper stage of the tool and the vacuum is applied. When the temperature of the substrate and the mold are equal and reach the preset level, pressure is applied for a certain period of time. The next stage of the process is cooling the substrate and mold sandwich under the same pressure. The final step of the process is separation of the master mold and substrate, now containing the embossed microstructures.

2.4 Confocal Measurements

To evaluate results of hot embossing experiments a laser scanning confocal microscope (LSCM) (Olympus Pty. Ltd., OLS1100) was employed. This microscope allows measurement of depth of the microstructures with high resolution (0.01µm), due to its very short depth of field. With this equipment non-contact 3D surface data acquisition is possible. It can be used as a common microscope in order to get 2D images of the surface. The advantage of a confocal microscope in comparison to other 3D information systems like interferometers, or fringe projections is the possibility to reproduce quantitative information from surfaces with a range of roughness, high aspect ratios, and varying reflectivity. Filtering and levelling operations are provided to post process generated image data. The quality of these images and the time taken to acquire images is in between a scanning electron microscope and an optical microscope. In Figures 3 and 4, the 3D images of a master mold and embossed channel in PS substrate are shown. Figures 4 and 5 represents 2D images of master mold and hot embossed channel respectively. In Figures 5 and 6, a black area can be seen running top to bottom, along the centre of the image. In these areas no texture and height information was acquired due to the steep wall angle of the nickel structure, and the height of the structure shadowing the surface area next to it. Because of this the full light intensity does not reach the bottom of the shim and moreover reflected light from the bottom is reflected again at the line structure so that the confocal microscope is not able to detect it. In Figure 7 a cross section profile is shown, and was measured on the image of the mold insert shown on Figure 5. The black areas on Figure 5 appear as noise within the graph. This graph was filtered in order to reduce the noise but this only removed high frequency components, leaving artefacts on the data. Filtered and unfiltered data show the same top and bottom dimensions so that the uniformity of the line in a small range can be declared. Moreover the width on top of the structure can be assumed to be the real value as there are no disturbing structures to influence the measurement. This assumption was made for all measurements.
Figure 3 3D image of nickel master mold, showing the 173μm wide bottom, and 200μm height. The features on the steep walls are artefacts caused by the measurement process.

Figure 4 3D image of an embossed channel in polystyrene using the shim shown in Figure 3. The embossed structure is 199μm deep and 172μm wide at the top.

Figure 5 2D image of nickel master mold as shown in Figure 3. The line shows the position of the cross-section measurement shown on the Figure 7.

Figure 6 2D image of polystyrene embossed channel, as shown in Figure 4. Dark areas are the steep side walls which do not reflect light back to the detector.

Figure 7 Master mold cross section profile, showing overlaying of filtered and non filtered data. The filtered data still shows artefacts on the steep side walls, but the width of the top and bottom features are accurate and unchanged.
2.5 Design of experiments

The main advantage of DOE method is simultaneous variation of all input parameters and representation of results in the form of second order equations. These equations provide the dependence of chosen response functions on input parameters.

In order to find the optimal operating parameters of the hot embossing process the Central Composite Second-Order Rotatable Design of experiments was used. This class of designs allows not only minimise the estimates of individual coefficients but also the acquisition of the constant variance in all points equidistant from the centre of design [13,15]. For polystyrene the maximum and minimum temperature parameters were determine as 85°C (Value 1) and 110°C (Value 5). Pressure on the sample is in the range from 10 bar to 200 bar and time (determined as time under constant pressure during heating phase of the process) was chosen in the range of 4 seconds to 20 minutes. As a result of planning 20 experiments were formulated. The cross-section area of the channels was chosen as the response function (as this is an important factor of estimation of productivity of microfluidic channel). Table 2 summarises code values and processing conditions of hot embossing process according to results, given by the software.

<table>
<thead>
<tr>
<th>Code values</th>
<th>Value 1 (-1.682)</th>
<th>Value 2 (-1)</th>
<th>Value 3 (0)</th>
<th>Value 4 (1)</th>
<th>Value 5 (1.682)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature, °C (X₁)</td>
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<td>90</td>
<td>97.5</td>
<td>105</td>
<td>110</td>
</tr>
<tr>
<td>Time, sec. (X₂)</td>
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<td>125</td>
<td>302</td>
<td>479</td>
<td>1200</td>
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<tr>
<td>Pressure, bar (X₃)</td>
<td>10</td>
<td>49</td>
<td>105</td>
<td>161</td>
<td>200</td>
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</tbody>
</table>

Table 2. Code values and process conditions for hot embossing process according to DOE method for input parameters as temperature, time and pressure.

3. RESULTS AND DISCUSSIONS

3.1 Embossing of PS

According to the design, experiments were carried out and every sample was then measured on confocal microscope. Cross-sectional areas of each sample were calculated and the analysis of results, including verification of model adequacy and significance of model coefficients, was carried out using software package “Design-Expert” ver. 6. The fractional error between cross-section area of the mold insert and cross-section area of the embossed channels was considered as the criteria for optimisation and was calculated using the following equation:

\[ Y = 100 \times \left( \frac{\left(A_{\text{mold}} - A_{\text{Replica}} \right)^2}{A_{\text{mold}}} \right)^{1/2} \]

Where:
- \( A_{\text{mold}} \) is the cross section area of the master mold structure in \( \mu m^2 \)
- \( A_{\text{Replica}} \) is the cross section of the embossed channel in \( \mu m^2 \)

Using the coded factors, the model can be expressed with the following equation:

\[ Y = 6.2 - 16.1X_1 - 12.97X_3 + 7.27X_1^2 + 7.52X_3^2 + 12.98X_1X_3 \]

In order to find only optimum values of \( X_1 \) and \( X_3 \) it is necessary to solve the set of the following differential equations:

\[ \begin{cases} \frac{\partial Y}{\partial X_1} = 0 \\ \frac{\partial Y}{\partial X_3} = 0 \end{cases} \]
Differentiation gives the set of two simultaneous algebraic equations with the following solution:

\[ X_1 = 0.75 \]
\[ X_3 = -0.4 \]

Therefore the optimum parameters are calculated to be:

\[ \text{Temperature} = 97.5 + 7.5 X_1 = 103 \degree \text{C} \]
\[ \text{Pressure} = 105 + 56 X_3 = 83 \text{ (bar)} \]

As the result of the DOE method for optimisation of hot embossing parameters it has been found that optimum temperature and the optimum pressure for PS substrate material are 103\degree C and 83 bar respectively. According to the software, the time factor plays an insignificant role. However, experimental observation shows that the holding-pressure-time in a range of 5-6 min gives better quality for microchannels 200\text{µm} deep and 173\text{µm} wide.

### 3.2 Embossing of PC

The aim for this work was the characterisation of the hot embossing process and the optimisation of process parameters such as temperature, pressure, time. The chosen material in this case was polycarbonate due to its widespread application possibilities. The master mold with double lines was used for this study. Dimensions of the microstructures are shown in Table 1. Line 1 and line 2 will be referred to as shallowest and deepest respectively. The confocal profile of the shallowest line is much clearer since the microscope is able to acquire data with a good signal to noise ratio. The aspect ratio of the deepest line results in a loss of data in the steep regions of the structure due to reasons explained earlier.

<table>
<thead>
<tr>
<th>Temperature, ºC ((X_1))</th>
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<th>Pressure, bar ((X_3))</th>
<th>Response Area, % ((Y))</th>
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<td>302</td>
<td>105</td>
<td>7.16</td>
</tr>
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Table 3 Range of parameters variation and fraction error of cross section area of the microchannels.

The confocal profile of the shallowest line is much clearer since the microscope is able to acquire data with a good signal to noise ratio. The aspect ratio of the deepest line results in a loss of data in the steep regions of the structure due to reasons explained earlier.
3.2.1. Pressure variation at 150°C

By applying only the vacuum pressure to the sandwich a relatively shallow surface shape is formed. Not surprisingly, by increasing the pressure the embossing quality becomes better and finally the corners are nearly filled. Thus a higher pressure influences the embossing quality positively. However, care should be taken not to apply too much pressure as this can damage the microstructures on the nickel shim, especially if the substrate temperature is below Tg.

![Figure 8](image.png)

*Figure 8 Effect of pressure variation at 150°C for the shallowest microchannel embossed in PC.*

![Figure 9](image.png)

*Figure 9 Effect of pressure variation at 150°C for the deepest microchannel embossed in PC.*
Increasing the temperature up to 150°C provides a significant increase of the embossing quality. Within the shallowest channel the results shown in Figure 8 are nearly perfect except the embossed structure for ~1bar. Slightly different are the results derived from the deepest structure, which are depicted in Figure 9. It can be observed that for all pressures above 1 bar, the maximum depth of the structure is achieved. However, the replication of the top surface and corresponding corner features is not achieved until pressures exceed 60 bar. In order to get better results the temperature or the hold time should be increased slightly as the corner is nearly filled. Thus a pressure of at least 30 bar is advisable in order to get good results with an acceptable temperature of 150°C for the shallowest aspect ratio, and the pressure must be increased to 60 bar for the highest aspect ratio structure with a short time frame of 5 min.

3.2.2. Temperature variation

The first test trial to show how different temperatures influence the embossing result was executed with an applied pressure of 75 bar. By applying 160°C over a time frame of 5 minutes the polycarbonate spreads beyond confines of the embossing shim, and the substrate becomes attached to the embossing tool. In order to prevent this, the pressure level was reduced to 33 bar for every cycle. The experiments were executed in two runs. The first run with temperatures from 140°C to 147°C and the second run with temperatures from 150°C to 162°C.

![Figure 10 Temperature variation at 33 bar using the shallowest channel microstructures.](image)

Depicted in Figure 10 are the results of the shallowest channel. It is noticeable that the embossing results above a temperature of 150°C are good and complete filling of the corner edges is obtained. A Savatksi filter, used to improve the data acquired by the confocal microscope, created the small peaks at the top corner of the embossed structure as this filter inflates sharp corners. Subsequent application of a mean filter further improves the data by reducing the noise, but does not remove the peak introduced by the first data filtering process.

Examination of the lines for 147°C and 150°C appears to show a paradox phenomenon by suggesting the lower temperature yields a better embossing quality. The same effect is seen in Figure 11 and is attributed to the fact that the experiments were executed in two runs and the 150°C cycle was the first cycle of the second run when the embossing system may have been uniformly heated.
3.2.3. Time variation

The time variation is the different duration of the hold time when pressure and temperature are kept constant. Zero minutes means that the pressure was applied and the cooling air was turned on the next moment. The results given in Figures 12 and 13 show the results for the shallowest and the deepest channels respectively. Due to the pressure and temperature settings the embossed quality decreases with increasing structure size if the applied time is the same. This is indicated by a less filling of the top polycarbonate corners and thus bigger areas with curved polycarbonate surfaces. On all of these images it can observed that a hold time of 0 minutes does not lead to good results not even with a quasi extended hold time by slow cooling. By using the parameters 150°C and 33 bar pressure, the time frame of the hold time for good embossing qualities reaches from 2 minutes to more than 10 minutes for deeper structures like deepest channel.
4. CONCLUSION

An experimental program was designed to optimise operating parameters, such as temperature, pressure and cycle time. A scanning laser confocal microscope was used to measure and evaluate the quality of embossed microstructures, and showed that useful measurement can be made, even for higher aspect ratio structure. At optimal conditions the difference between dimensions of replicated microchannels and inverse structure on master mold was in a range of 1% for PS substrate material and 4% for PC substrate material. The optimal conditions for embossing microstructures into polystyrene are: 83 bar pressure on the sample, 103°C temperature of the mold insert and the substrate, and 5-6 min hold time during the heating phase of the process (microchannels 200μm deep and 173μm wide). For polycarbonate the optimum parameters are: 155°C temperature and pressure is in range of 30 for the shallowest microstructures and 60-91 bar for deeper structures. Holding pressure time during heating phase of the process is 2 min for the shallowest microchannels (14μm deep x 28μm wide) and 10 min for deeper channels (85μm deep x 38μm wide).

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