Influence of process parameters on edge replication quality of lab-on-a-chip micro fluidic systems geometries

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Published in:
ANTEC 2013 Conference Proceedings

Publication date:
2013

INFLUENCE OF PROCESS PARAMETERS ON EDGE REPLICATION QUALITY OF LAB-ON-A-CHIP MICRO FLUIDIC SYSTEMS GEOMETRIES

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Abstract

The growing demand to manufacture, with high accuracy, structures enabling transportation, treatments and measurements of minuscule biomedical samples on polymer substrates is pushing the process capability of technologies such as injection molding to their limits. To characterize and asses the replication quality of molded micro-features on cyclic olefin copolymer (COC) a tool insert collecting critical channel cross sections was manufactured. The master was made by UV lithography and subsequent nickel electroplating. Effect of packing phase parameters (packing time, packing pressure) and mold temperature were investigated. Moreover, consequences of different positions, directions and nominal channels width were considered. Edge replication quality was quantitatively characterized, analyzing calibrated scanning electron microscope (SEM) images with a digital imaging processing software. Results showed better replication fidelity mainly influenced by the higher mold temperature and also by higher packing pressure, whereas poor edges quality was observed for the smallest replicated test structures.

Introduction

During the last decade the fields of micro and nanotechnology, covering a large number of disciplines, have experienced a dramatic growth in or towards commercial exploitation routes. In particular the possibility of producing miniaturized analytical systems driven by the need of cutting costs, reducing the consumptions of expensive reagents and by increasing throughput and automation have attract industries interest [1]. The rapid expansion of these new microfluidic systems has led to further development of polymer mass fabrication techniques to pattern polymer components [2, 3]. High accuracy in terms of manufacturing precision and resolution are the main issues to decrease the existing technological gaps enabling Lab-on-a-chip (LoC) prototypes to reach the market. Recently these prerequisites for achieving successful device functionalities are enabled by the possibility of combining state-of-the-art polymer replication techniques and technologies such as UV lithography, nano imprint lithography and e-beam lithography to produce micro and nano features [4]. Hence the development of these analytical systems passes through consistent manufacturing systems that determine reliability of the production route. Hot embossing, injection molding and micro injection molding are popular methods to replicate micro features [3, 5]. However, both methods face challenges in filling/replicating the polymer melt into the micro and sub-µm geometries [6]. The correlation of the replication accuracy to different process parameters is a complicate issue and it has been extensively investigated in literature [7, 8]. Being mold temperature one of the most important parameters enhancing the quality of replicated micro feature, electromagnetic induction heating technology combined with water cooling systems has also been introduced [9]. In addition vacuum and venting concepts have been tested [10, 11] to evaluate their contribution to air escape during the injection phase especially at high speed. Reported results [12, 13] for injection molding of micro and sub-µm positive structures with optimized processing conditions using vacuum venting showed little to no effect on feature definition. The important issue of the presence of round corners on the bottom of the tooling channels due to the thermoplastics tendency of releasing large quantities of vapor turning on being trapped air and premature solidification of the polymer melt have been reported [14]. A direct consequence of poor edge replication quality of the polymer channels is the not-effective thermal bonding between the chip substrates and the relative polymer lid which would deform excessively into the channels even using low bonding temperature and load [15, 16].

This research investigates the effects of three process factors (mold temperature, packing pressure and packing time) on capability of replicating three different aspect ratios (1/20, 1/4, 1/1) produced test structures equivalent to critical channel dimensions on the real LoC prototype. Moreover effects of features orientation and position in respect to the polymer flow direction were studied. Relative and absolute deviations from full features replication have been quantified by means of calibrated SEM images. Measurements repositioning on the different produced samples gave the possibility of comparing results and effect of the designed experiments. The present paper addresses one of the key challenges in the manufacturing of microfluidic devices, identified in their thermal bonding with cover plates or foils, with the need of reliable measurement able to quantify the replication

quality of channels top edges. Furthermore the work contributes to develop and establish standard rules for LoC design, fabrication and in line process quality control.

Master making

The master was fabricated using a 365 nm UV mask aligner process with 2 mask layers on a 100 mm silicon wafer. Both layers were defined in AZ5214E photoresist and dry etched using a reactive ion etching (RIE). The etch depth was controlled by etching time. Once the micro structures were etched into the silicon master, a titanium/ nickel seed layer with a thickness of 10 nm and 100 nm respectively, was deposited by sputter deposition prior to electroplating of a 320 µm nickel layer.

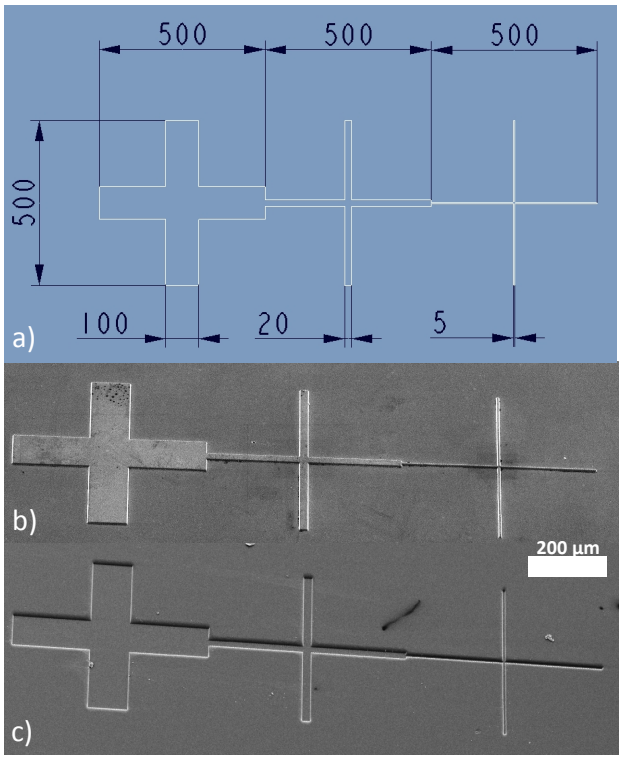

Figure 1: Test structure geometries: a) Technical drawing shim geometries with the relative quoted nominal crosses dimensions (all the dimensions are expressed in µm); b) nickel shim produced geometries; c) COC test structures polymer replica.

The mirroring of the structures during the replication process adds the complication of measurements relocation over different sample replicas. Moreover LoC devices are most of the time representing complex miniaturized analytical systems implying different aspect ratios of the designed geometries. Therefore to overcome part of this measuring challenges test geometries representing some of the critical dimension of a sub-µm fluidic system for optical mapping of genomic length DNA were manufactured. The so called "process finger print" is composed by the test geometries shown in figure 1. The three produced crosses have constant height (nominal value on the silicon master equal to 5 µm) with horizontal and vertical wings width of 100 µm, 20 µm and 5 µm respectively. The crosses are positioned in the center of the produced 22 × 22 mm^2 nickel shim used for polymer injection molding.

Micro injection molding

Micro cross geometries have been replicated on polymer substrates by injection molding. Replication quality of the three dimensional features have been quantified in order to link the change in process parameters with products accuracy. A statistical designed set of experiments specifically aimed at studying and characterizing the channels edge definition, was carried out. Three process parameters, mold temperature, packing pressure P(pack), packing time t(pack) were varied. Replication quality effects on the projected polymer structures were evaluated considering melt flow direction in respect of feature position, orientation and their nominal widths. Every time process parameters were changed, up to 10 pre-test samples were injected and weighed to ensure equal part filling conditions. Afterwards, process repeatability was assessed to be equal to 0,8 mg weight standard deviation over 15 reference parts. Process levels were set according to the following considerations:

- Mold temperature is the parameter that can facilitate the even distribution and packing of the features and was kept constant using oil coolant. Mold temperature of 120°C (min) ensured test structure replication and filling at the fixed high injection speed. Mold temperature of 130°C (max) was set as a consequence of the fact that even though from the manufacturer recommendation is stated that mold temperature can be set to almost 150 °C already at 140°C burnt edges of the samples were observed.
- Packing pressure was fixed at 400 bar (min) and 600 bar (max) considering a tradeoff among effective shrinkage compensation, dimensional accuracy and automatic demolding without distortions of the final geometries.
- Packing time of 3s (min) and 4,5s (max) were established during pre-experimental tests as levels ensuring effective shrinkage compensation, optimized stable weight and dimensional stability and absence of flash in the parts.

Generally, process parameters were set to optimize the resulting quality of the produced geometries and considering machine capability, material physical

properties and process feasibility. Melt temperature was chosen in accordance with the material supplier specifications, as a balanced value to avoid material degradation and premature solidification during the filling of the cavity. Finally, high injection speed was selected to enhance replication, bearing in mind the possibility to yield, with a too high speed, high internal stresses and air capture.

Table 1: Process parameters

Process parameters	Values
Melt Temperature [°C]	250
Injection Speed [mm/s]	150

In this study, the focus has been on the replication of the transparent polymer COC Topas 6015 substrate which has characteristic compatible with biomedical requirements. Different COC grades are being used in life science applications due to their excellent optical properties, low moisture absorption and high resistance to degradation from most bases and acids solutions. The injection moldings were executed on a full electric injection molding machine with a reciprocating screw diameter of 18 mm and clamping force of 600 KN.

Measurements Strategy

Scanning Electron Microscopy (SEM) providing high resolution imaging with high depth of focus for characterization of critical dimension measurements has been employed to characterize nickel mold features and injection molded COC channels, see figure 2. Pixel size of the SEM images was calibrated for nominal magnification levels in the range from 100x-10000x at fixed working distance [17].

The different samples were aligned and positioned on the SEM stage after being sputter coated with 20 nm thick layer of gold to enhance the detection of several emissions coming from the interaction between the scanning electron beam and the different specimens.

Figure 3 summarizes the different measurement locations were SEM images were taken for later analysis using commercial available scanning probe image processor software (SPIP) [18].

In order to quantify the distance of the polymer front flow to the channels edge, distance measurements in respect to the wings direction were carried out. A conversion of image pixels into dimensions was possible due to the SEM picture calibration, carried out by digital imaging post-processing.

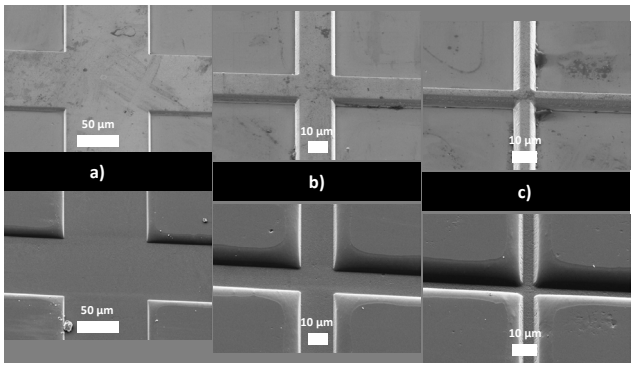

Figure 2: Effect of draft angle on final replication quality of the produced polymer sample. Top: nickel insert; bottom: corresponding COC replicated geometries.

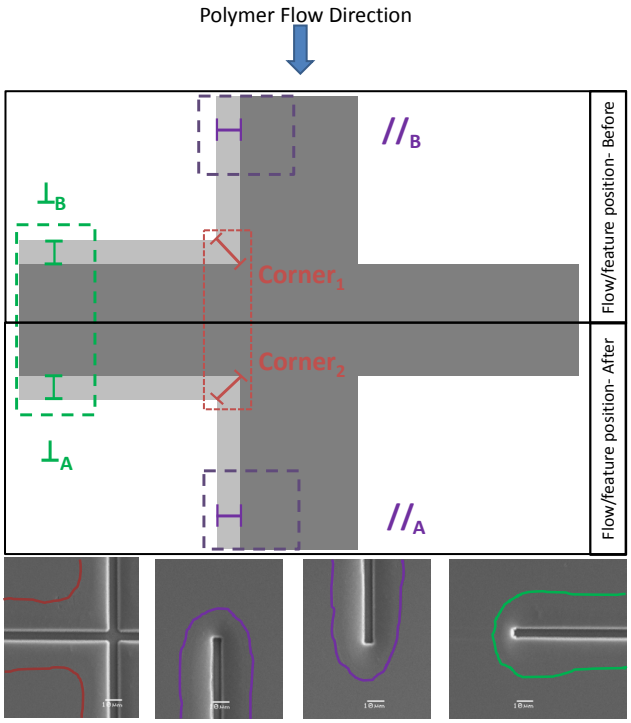

Figure 3: Measuring strategy scheme. Top: different measurement locations for each single produced cross; bottom: example of measured geometries on the polymer part, different colors correspond to the different measurement locations depicted on the measurement strategy illustration.

In particular, a measuring routine to quantify the diagonal distance in proximity of two wings intersection has been adopted. To ensure measurement reproducibility of the non-conventional measurand a method based on fast and precise alignment was necessary. Therefore a 45° line from the adjacent vertical channel wall was traced for every single measured corner. This ensured for all the measurements a reference direction to evaluate the process capability on replicating critical geometries. The traced line allowed the generation of a single point

intersection between two lines perpendicular to both corner edges, see figure 4, and thus the determination of the distance of the flow front to the corner.

Measurement results have been expressed as average value of three different measurements for all the different positions in all the produced polymer crosses. Measured results of the relative unfilled channel distances where calculated as ratio between the absolute unfilled distances and the different trances width (1).

$$Rel.unfil.channel = \frac{Unfilled\ dist.}{Nickel\ trances\ width} * 100\% \qquad (1)$$

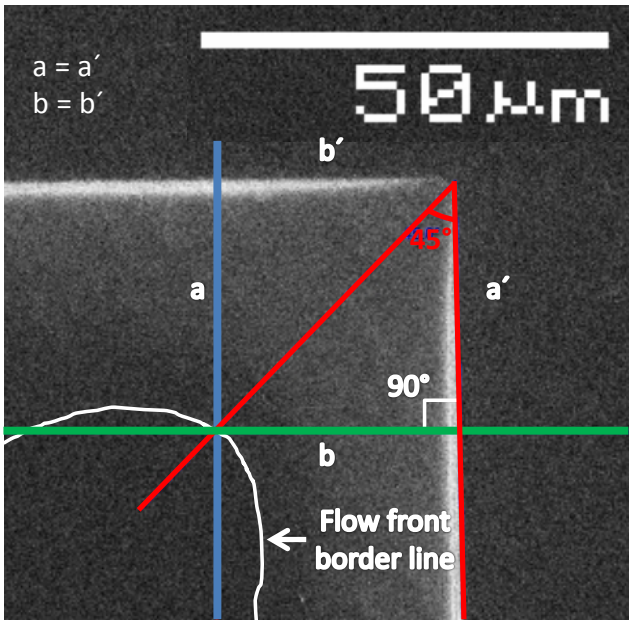

Figure 4: Proposed measuring method to quantify diagonal distances on analyzed high quality SEM pictures.

Statistical analysis and discussion

To evaluate the accuracy of the process subject to different process conditions the absolute and relative unfilled channels width are expressed to evaluate the variation of replication fidelity of the channels edges. The experimental results are shown in the format of main effect plot for the two previous mentioned output quantified in micrometers (see figure 5) and percentage, (see figure 6) respectively.

The graph in figure 5 shows that the replication fidelity is mainly enhanced by the higher mold temperature maintained along all the phases of the molding process, introducing longer overall cycle time for conventional mold designs. High packing pressure also increases the edges definition of the channels being this the parameter capable of pushing the polymer flow further before its complete solidification and to adhere on master protrusions walls, edges and corners. Moreover the analysis shows process limitations on replicating sharp corners (i.e. higher unfilled distances are observed for the diagonal direction) confirming the difficulty of evacuating the produced vapor of the thermoplastic substrate from such closed corners. Due to the high injection speed and thus the short injection time (< 100 ms) the polymer flow is not influenced by the crosses wings positions, showing no evidence of decrease of edge replication from before to after position (see figure 3). Mean value of unfilled distances variation from the full edges replication decrease 5 µm with decreasing channel width.

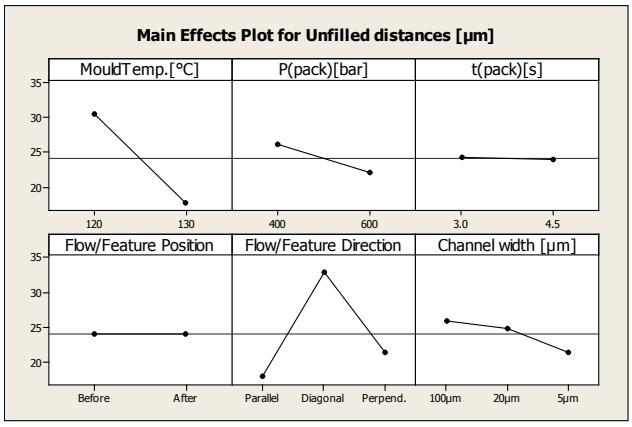

Figure 5: Main effect plot for unfilled distances.

In order to quantify and understand how the rounding of the polymer channels top edges relates to their decreased absolute widths, results obtained applying (1) are analyzed and presented in figure 6. An overall flattening of the different trends, due to the data normalization is observed. Nevertheless, data means of high mold temperature and high packing pressure contributed to increased replication fidelity. Notably, a significant increase in unfilled percentage of the channels edges relatively to the smaller channel width was measured, showing the challenge of replicating channels with increasingly smaller width.

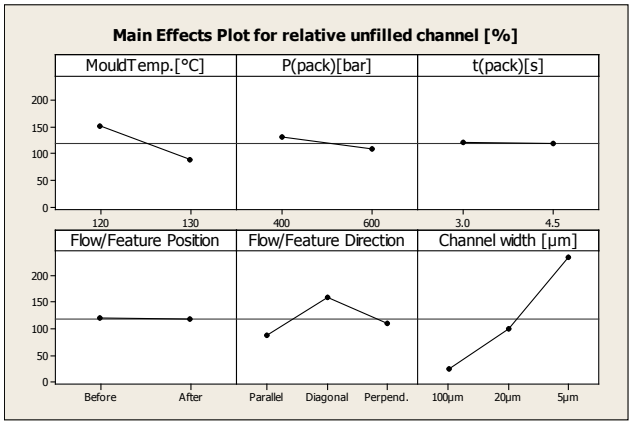

Figure 6: main effect plot of relative unfilled channels distances

Conclusion

Critical geometries of a microfluidic system used for optical DNA mapping were produced as a reference to evaluate and asses critical factors affecting the replication quality of polymer parts with focus on edge channel definitions responsible of effective thermal bonding between the chip substrates and polymer lids. Replication quality was quantified in terms of absolute and relative, unfilled distances to the structures dimensions. A measurement technique based on high resolution imaging obtained from scanning electron microscopy was employed.

Based on the statistical designed set of experiments, replication fidelity of the nickel protrusions has been measured on the corresponding polymer projections. Unfilled distances decreased in combination of high mold temperatures and high packing pressure. Replication of crosses corner edges results on being the most difficult feature to be reproduced. Unfilled channel edges distance increases almost linearly with the downscaling channel width.

Future development are warranted to achieve better channels edge definition through a improved trapped air and vapor evacuation yield by vacuum venting system and in-line material drying. Moreover, being mold temperature the most effective process parameter on surface replication, variotherm technologies enabling rapid mold temperature variation are needed.

Acknowledgments

The present research was carried out within the National Danish Strategic Research Center POLYNANO (http://www.polynano.org/) supported by the Danish Council for Strategic Research.

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