Process Control in Micro-Embossing: A Review

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Abstract— A promising technique for the large-scale manufacture of micro-fluidic devices and photonic devices is hot embossing of polymers such as PMMA. Micro-embossing is a deformation process where the workpiece material is heated to permit easier material flow and then forced over a planar patterned tool. In this work we review the basic process and the state of research with respect to manufacturing process control, where the latter is defined as methods for minimizing variation in the product while maximizing production rate. From this review we conclude the following: Several investigators have reported success at creating micron scale features using this process, but none have performed a formal characterization or optimization of the process.

I. INTRODUCTION

THE process of embossing of thermoplastic materials is not a new one, but it is receiving renewed attention as a good candidate for high volume production of micro-fluidic and micro-optical devices. It is a single step process for net-shape forming of components that might otherwise require multiple processing steps using conventional lithographic processes on glass or semiconductor substrates. One of the key materials for such products, polymethylmethacrylate (PMMA) has both good processing characteristics and good function characteristics. In the latter, it is favored for both optical clarity and fluid compatibility. This review will focus primarily on PMMA.

Microfluidic devices are microsystems that control the flow of small volumes (nano- or pico-liters) of fluids (gases or liquids) through micro-capillary channels on a "chip" that is integrated with computerized analytical instruments. Such devices contain active and passive microstructural features that control the flow and mix of the fluids to produce physical, chemical and microbiological reactions on small volumes of materials. They are beginning to be widely used to conduct biomedical research and to create clinically useful technolo-

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gies. Such devices hold the promise to minimize the time and costs associated with routine laboratory experimentation and analysis, and have potential applicability in many areas including the pharmaceutical, genomic, chemical, and diagnostic industries.

This presents the potential for a large yet diverse market for the basic microfluidic "chip". This in turn creates a need for a manufacturing infrastructure that can produce such chips in high volume with extremely high quality. While there has been a growing amount of work examining the basic feasibility and process limits for embossing of features at the micron scale, there has been little published in the area of manufacturing and more importantly in the field of process control. In the following, the area of process control will be carefully defined in the context of micro-embossing and this description will be used as a template to evaluate the current state of research.

II. TARGET APPLICATIONS

There is considerable literature regarding the use of embossed components in micro devices, and they tend to fall into the following broad categories:

- · Micro reactors
- Micro fluidic flow systems for physical separation
- Micro optical devices

In all cases the product performance depends upon creating patterns or features in a polymer workpiece that have characteristic dimensions in the 0.1 to 100 μm range. There is currently a move to net shape forming (e.g. embossing and injection molding) of polymers for applications in the biomedical field. As pointed out in [1] and [2], this is motivated by the prospect of high production rates and lower costs when compared to the traditional methods using glass or silicon and lithography based methods. The attributes of embossing are particularly relevant in the biomedical field, where disposability is important and the customer base large and repetitive. In addition, many polymers (such as PMMA) have good biocompatibility and favorable optical properties. As a result, there have been many recent publications looking at the use of embossed components in the above application area.

A. Micro fluidic bio reactors and separators

One significant application realm is microfluidic reactors and separators. Such systems allow chemical analyses to be

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performed with pico-liter volumes of reagents, facilitating many tests with same sample volumes, and rapid reaction rates. They also allow numerous tests to be performed in a very small area, in a device that can be disposed of after use.

Pumera et al. [3] discuss the use of PMMA based chips for electrophoretic separation and report excellent performance. More relevant to this review, they conclude that if the manufacturing base for such devices can be created, single use applications will become broadly viable. Krishnan et al. [4] similarly use a micro fluidic device for gene expression analysis, combining separation and reaction in a single device. While focusing on the function of these devices, they note that the key to wide spread use is simple, low cost fabrication of complex patterns in appropriate materials. Similarly Ricco et al. [5] discuss the use of microfluidic arrays for applications in both DNA analysis and in "high-throughput pharmaceutical discovery". Again, they extol the virtues of polymer–based devices for their low cost, high volume, high quality potential.

The concept of continuous flow bio-assays is discussed by Williams *et al.* [6]. In their application, they use micro channels to mimic the effects of human capillaries. Of particular interest here is their observation that the performance of these devices depends on low variability in flow channel dimensions from chip to chip. The resulting variable pressure drops could result in unacceptable flow variations. They present a specific measurement that shows that current manual prototyping method can lead to normalized standard deviations in pressure drop as high as 8.5%. This is one of the few investigations that have related performance to product quality.

Another vote for polymer-based devices is given by Schulte *et al.* [7] who discuss commercial devices for sample preparation and hematological analysis. They use injection molding rather than embossing to create parts with channels and reservoirs, where the flow is driven by both hydrostatic forces and by gradients set up with internal absorptive materials. Another commercial device described by Cronin *et al.* [8] is used for DNA analysis and drug discovery, and is made using either injection molding or embossing. However, they do not delineate the performance difference between the two.

A third commercial venture in microfluidic chips is described by Preckel *et al.* [9]. This system detects cellular fluorescence after mixing the sample with various reagents. They report good correlation with macro scale tests, but their devices were all fabricated from glass substrates.

Another method of driving flows is to spin a CD sized chip embossed with micro channels. This method is described by Wenner *et al.* [10] and they created their "CDs" using several methods including embossing of PMMA and lithography of SU-8. Madou *et al.* [11] also report the use of hot embossing of PMMA and other materials for disk-based micro fluidic devices and also compare this to injection-molded devices. In general is it found that injection molding has faster cycle times while embossing produces a more uniform product.

B. Optical Components

Micro patterning of surfaces has been used for some time to create surfaces with controllable reflection and diffraction characteristics, and hot embossing of smaller and smaller features is receiving attention. Tuteleers *et al.* [12] compare the results of using vacuum casting and hot embossing. They report that replication with both is not perfect and conclude that careful process control will be necessary to achieve the required optical properties in the product. Hot embossing is also used by Lin at al. [13] to create micro-pyramids for brightness enhancement materials for LCD displays. They report on the use of PMMA and PVC for features as small as 2 µm. They demonstrate that good performance can be achieved with embossed polymers, but in a companion paper discussed below, (Lin *et al.* [24]) they point out large discrepancies between lab scale results and full-scale commercial forming results.

The use of nano-scale patterning on optical surfaces to reduce reflection is reported by David *et al.* [14]. Using quartz tooling and embossing of polycarbonate sheets, they created patterns with channels spaced at 0.12 μ m. The resulting surfaces demonstrated significant reduction in reflectivity over wavelengths of 250 – 550 nm.

Newer optical applications are beginning to be explored that combine microfluidics with photonics to create high-speed switches and light modulators. While this work is in early stages, some applications have shown the potential for using devices patterned by micro-embossing methods. For example, the work of Mach *et al.* [15] describes the development of optical fibers that can be tuned using microfluidic plugs. This can be extended to creating microfluidic devices with multiple channels to provide high speed switching (~100ns.) and narrow band filtering. A similar result is reported by Kerbage and Eggelton [16] for creating tunable gratings within optical fibers.

III. MICRO-EMBOSSING

The process of hot embossing of thermoplastic polymers involves the plastic flow of material around a tool that has a shape inverse of the desired part shape. The material is first heated to a point between the glass transition temperature (T_g) and the melting temperature (T_l) and then the tool is pressed into the material uniaxially. (It is the polymeric equivalent of a hot forging process.) This sequence is shown schematically in Fig. 1. A corresponding lab-scale set up for embossing is shown in Fig. 2. This setup includes heated platens, tool and workpiece holders, and a single axis actuator for applying a well controlled forming forces.

The success of this process is measured both by the dimensional accuracy of the final part and the presence of residual stresses that may affect performance. Depending on the application, both feature dimensions and surface finish will be of concern.

The process proceeds by first heating the material and tool and then applying a forming force. As this force is applied, the material begins to flow into the tool, initially flowing across the boundary of the tool features. Once complete filling of the features has occurred, the forming pressure then held for a sufficient time must be allowed for the inherently viscoelastic material to flow into the tool, This hold time is a func-

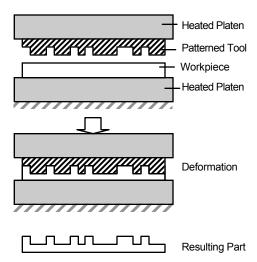


Figure 1 Basic Steps in Embossing

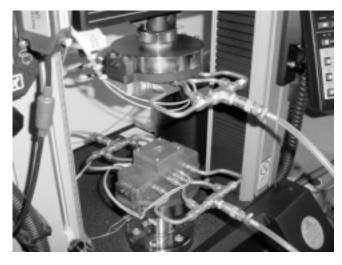


Figure 2 A lab-scale embossing setup at MIT

tion of both material temperature and forming force. In the end it is this time – force tradeoff that is crucial to the success of this process in a manufacturing environment, where both quality and production time are vital. The equipment variables that are available to control this process include: tool force trajectory, tool velocity trajectory, tool and platen temperature, hold time at maximum, and the cooling rate of tool and platen. The time history of these variables is shown schematically in Fig. 3.

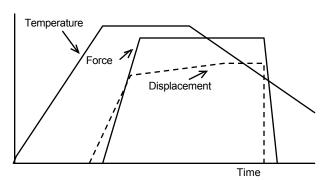


Figure 3 Time History of Process Inputs (With force control)

IV. MANUFACTURING PROCESS CONTROL

Process control is defined herein as the concern with final product accuracy and variation in a flexible, high volume production environment. It is an inherently feedback-oriented problem, with many loops operating at different time scales and for different purposes. However, the overall goal remains that of ensuring conformance to a set of design specifications for the product, typically in the form of key dimensions and material properties for the product.

The various loops that are invoked are shown schematically in Figure 4. A hierarchy for process control is presented that begins with a basic process characterization. Here the outputs are defined as key characteristic dimensions of the resulting workpiece and the properties of the material after forming. (Indeed all unit processes can be uniquely characterized by a set of geometry and property outputs). The inputs are those variables we can actively manipulate to achieve changes in the output, such as temperature, forming force, forming rate, hold time, etc. The characterization is a first order relationship between inputs and outputs, along with identification of disturbances or noise factors that can add to process variation. The next levels in the process control hierarchy are:

- Statistical characterization of the outputs with all inputs held constant
- Open-loop optimization of the process inputs to minimize error and variations
- Closed-loop control of the embossing equipment states to minimize effects of equipment variations
- Closed-loop control of the state of the workpiece to minimize effects of material variations
- Closed-loop control of the actual outputs to achieve onspecification products with minimum variation

These steps serve as progressively more aggressive attempts to improve process capability, i.e. the statistical performance of the process as compared to the design specification. In this paper we will review prior work in the field of polymer embossing, including both macro and microscale applications.

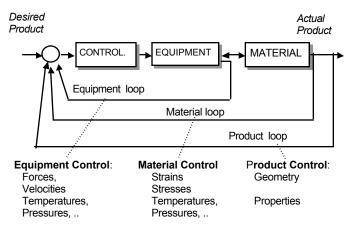


Figure 4 General Process Control Loops

V. . TOWARD PROCESS CONTROL: STATE OF THE ART

A. Process Characterization

Not surprisingly, with a new process of interest, most work in this field has been concentrated on the basic understanding of the physics of embossing at the micron scale. Under this heading we can expect work as generic as constitutive properties of PMMA through process mechanics, simulation, and finally specific empirical observations from forming tests. Most work has tended toward empirical studies, aimed at getting a hands-on feel for what the issues and relevant parameters are in this process. In many cases the fabrication work is subservient to device design and the process is treated as a one-off or prototyping one, rather than a robust production process.

An example of generic work is that of Kobayashi *et al.* [17] who present a basic study of material behavior above the glass transition temperature (T_g). Looking at shear deformation in several glassy polymers including PMMA, they observed a sharp change in viscosity at 1.05 T_g . When the material was annealed this transition point remained, but the viscosity change was lower in magnitude. This work was carried out at low strains and strain rates typical of embossing, but not relevant to injection molding.

Perhaps more specific to the micro-embossing process is the work of Heyderman $et\ al.$ [18]. They studied the flow of PMMA into micron scale cavities at various temperatures above T_g . The work included measurement of flow into 20 μ m cavities 170 nm deep at various stages of filling. As expected, the material tends to flow in first at the edge where the pressure gradient is the greatest, and fill toward the center as time progresses. They then used a 2-D squeeze flow analysis to develop a prediction of the time to fill a cavity. Their result is given by:

$$t_f = \frac{\eta S^2}{2p} \frac{1}{h^2} \tag{1}$$

where η is the viscosity (which [17] shows is strongly temperature dependent p is the net pressure, S is the inter-cavity distance and h is the net platen penetration into the polymer. Since h is a function of the volume to be filled, it can be rewritten as

$$h=A/S$$
 (2)

where A is the cross sectional area of the 2-D cavity being filled. If we assume that S and A are given by device requirement specifications, then the processing time lower limit is given by the inverse of the pressure and the viscosity. This analysis assumes pure adhesion (no slip) at the boundary and an incompressible material. It also assumes either a single cavity or that all cavities are of identical volume.

A qualitative companion study looked at how the material actually flows in embossing trials in 3-D cavities (Schift et al [19]). When observed at the micron scale, flow into cavities showed marked patterning, including pillaring, circular lines

within a cavity, and "star" patterns. These are attributed to the specific geometry of the cavity as well as to the effect of electrostatic forces. However, additional investigation is needed to fully understand these effects. Perhaps more important here, a strong fingering pattern was observed when forming rates are high. This is attributed to the effect of entrapped air and possibly residual moisture. Internal heating, with the resultant disturbance of the flow, from rapid shear of the material is also observed. Finally, viscous fingering was observed if the material was demolded before sufficient cooling had occurred, again presenting a limit to production rate.

The subject of entrapped air was addressed by Roos *et al.* [20]. They looked at various patterns and process cycles (time history of temperature, forming pressure, and vacuum) and found that deformation was more uniform and had fewer defects if a vacuum was used. The extent of this effect depended upon the molecular weight of the material, with less dense materials being more sensitive to entrapped air. This was particularly important for large tools (10cm diameter with 0.4 – 10 µm features). They made more detailed quality assessments, which will be discussed in greater detail below.

B. Process Quality Assessment

While the above research has been mostly empirical, they do not in general comment on the overall quality of the final product. Since the overall goal of process control is to regulate quality of the final product, it is vital to know the key characteristics and problems with these that can arise during the embossing process. Roos *et al.* [20] in their study of the effects of vacuum, created a set of quality criteria that allowed them to grade the effect of their experimental changes. By looking at:

- width of flow borders (in µm)
- area of "LISA" defects (self assembled trenches at the surface)
- viscous fingering (% of features present)
- visual uniformity (e.g. % degree of filling)

they were able to distinguish the performance of various processing regimes (especially the effect of vacuum). This work is significant in that it establishes a measurable set of attributes that can be used for statistical studies, process optimization and ultimately for active process control.

In a related paper, Roos *et al.* [21] investigated the use of a commercial embossing system, for making PMMA parts with features ranging from 0.4 to 100 µm on a large-scale (4in) workpiece. They also assessed quality based on the degree of filling (as opposed to specific dimensional variation). They identify temperature and hold time as critical to forming success, and imply that pressure needs to be above a certain threshold, but is the least sensitive variable of the three. Unfortunately, the quality assessment is rather qualitative, and serves only to support the conclusion that such mutli-scale forming over a large area is feasible.

Specific statistical data for micro-embossing is presented in Lee *et al.* [22] who are interested in bio-fluidic applications. Since they seek an inexpensive, disposable product, their concern is inherently related to true manufacturing, and accordingly, to process statistics over a large ensemble of products.

As opposed to the informal quality study in [20] they fabricated a number of identical devices with identical processing conditions to look for variation in specific dimensions, rather than more binary "defects". For example, they noted that over 10 separate parts, the surface profiles (including 100mm width channels 400 µm deep) had an average width of 102.9 µm and a relative standard deviation of 2.2%. The relative depth standard deviation was quoted as "less than 1%" on an average of 39.12 µm. They also characterized their etched quartz tool and concluded that reproduction is very good with microembossing. However, the dependence of these statistics on processing conditions (rate, temperature, pressure) was not investigated.

Bacon *et al.* [23] describe a series of embossing experiments with 300 - 500 mm features in PMMA. Using fixed hold time and no vacuum, they varied the embossing force and the temperature of the top and bottom forming platens from ambient to > Tg. An output data table is presented for a total of 21 different combinations of these inputs and the outputs are listed in terms of relative quality of pattern reproduction (similar to Roos *et al.* [20]). While only a start, this work is an example of the basic empirical characterization of embossing that can be done using some form of designed experiments. However, there was no attempt to determine a specific functional relationship between inputs settings and output quality.

In a paper that addresses directly the issue of "manufacturing environment", Lin et al. [24] compare the quality of devices made in a laboratory environment with those made in a commercial environment. In both cases they were concerned with forming 30mm micro-pyramids. The lab process used PMMA while the commercial used PVC. There was no attempt to tune the processes, and the main distinction (aside from the material) was the processing time. The lab process, given a total cycle time of 2 hours was able to reproduce the tool faithfully, while the commercial process, operating on a 1 minute cycle time, did not. This clearly emphasizes the effect of time on quality, and the need for a better understanding of processing condition – quality relationships when production speed is important. An interesting side note is that in both cases they noticed an increase in the roughness of the finished product over that of the tool, with the commercial process being 2-3 times greater that the lab process.

Finally, in Lin *et al.* [25], the problem of quality is addressed from a fundamental point of view, with an analytical and simulation study of shrinkage in hot micro-embossing of PMMA. While there is no empirical verification of the results, they do conclude that proper use of holding pressures during cooling can greatly reduce the amount of shrinkage, similar to common practice in injection molding.

C. Process Control

Most of the above work concentrates on with the creation of a single artifact to be used for functional studies, the basic mechanics of the process, or specific empirical studies on process feasibility. None have addressed the full production environment that is defined by time-critical, high quality requirements. While defects and variation in dimensions is acknowledged, there has yet to emerge a literature on how best to characterize the manufacturing quality-product performance relationships nor is there any work focused on optimizing the capability of this process. Based on the process control hierarchy presented above, it appears that the state of the art has progressed through some basic process characterization, wherein the critical variables for the process are identified, and some initial studies of consistency and gross production limits have been made. Thus it can be concluded that true process control work has yet to be done on micro-embossing, and should be focused on identifying viable strategies for control (ranging from simple SPC to sophisticated in-process feedback control) and relating these to ultimate performing limits.

Examples of active process control do exist in related fields, in particular in the fabrication of micro electromechanical devices (MEMS) from silicon substrates. Building on experience in the semiconductor fabrication arena, there are several illustrative examples of where micro-embossing manufacturing research needs to direct it attention. As the call for smaller characteristic dimensions and larger yield volumes continues, active control methods will continue to replace old methods and, reactive, statistical process control. To this end, Fisher et al. [26,27] propose using in-situ Micro-sensor arrays that can be used for "calibration, control, and monitoring of semiconductor manufacturing processes." These sensor arrays are an integral part of a wafer and can provide during-process information without the expense and complexity of external measurement stations. The authors provide experimental data taken during an XeF₂ etch cycle that includes both etch rate and thickness measurements taken at several locations. The data shows non-uniformity in both etch rate and temperature across the wafer [26]. Such proof of non-uniformity, in a process that is assumed uniform, shows a need for process improvement. This in-situ measurement approach has the advantage of having new sensors for each cycle, thus avoiding sensor maintenance problems.

As a classic example of in-process dimensional control, Bosch-Charpenay et al. [28] show a method for real-time depth measurement using light waves and interference patterns for deep reactive ion etching (DRIE). Because the measurement systems are machine-based, they do not suffer sensor erosion problems of Fisher et al. [26,27]. Schaper et al. [29] and El Awady et al. [30] introduce a full closed-loop system for process improvement. The authors present a combined bake/chill system for photoresist processing in the lithography process. Concentrating on the post-exposure bake step, the authors cite a need for temperature control to within 0.1C for temperatures between 70C and 150C. Traditionally, this step has been done with large thermal mass systems that are designed to maintain uniform temperatures even when subjected to "disturbances," such as a new wafer being placed on them. The authors present a combined bake/chill system to prevent the need for wafer transport and to improve temperature uniformity on the wafer, especially during transient warmup/cool-down cycles. The authors present a temperature uniformity improvement of 3C at 70C mean temperature for a 6 inch square, 0.250 inch thick quartz substrate.

VI. CONCLUSION

The explosion of interest in micro patterned polymers has been driven by numerous exciting applications in microfluidics, micro reactors and optics. While much has been done to establish the functional suitability of embossed product from PMMA and other materials, it is evident that the manufacturing implications of these methods have not received careful study. We conclude from this review that a program focused on the key process control issues of quality and rate limits is critical to realizing the full potential of these emerging technologies.

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