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TECHNICAL PAPER

Technology of microthermoforming of complex three-dimensional parts with multiscale features

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Abstract Thermoforming parts with micro-scale design features require use of thin polymer films. Low heat capacity and fragility of thin (<100 μm) polymer films requires new technical developments for precise heating and form pressure application. This paper will present results of an improved process based on a new replication machine. Temperature stability and pressure distribution is highly improved, even at reduced cycle times. With an upgraded temperature range microthermoforming of high temperature polymers is possible. This technology is well suited for the replication of three-dimensional micro parts with multiscale features. Replication of hollow parts with multiscale features is also possible. Outer dimension in macroscopic length scale and functional design features in microscopic length scale are combined with nanoscale surface structures in an affordable technology.

1 Introduction

Thermoforming and blow moulding are well established replication technologies. Thermoforming as a replication technology of thin walled parts is mainly used for the fabrication of enclosures, packages and large-scale parts. Thermoforming is a manufacturing process where semi finished products, in kind of thermoplastic polymer films or plates are heated to softening temperature and formed to a specific shape into a mould (Throne 2008). Preshaping can be done by mechanical stamps, whereas main shaping process is done by gas pressure differences. A high pressure micro thermoforming process is used to fabricate film microchips for capillary electrophoresis (CE) (Truckenmüller et al. 2002) and for three dimensional cell cultivation (Truckenmüller and Giselbrecht 2004) at the Forschungszentrum Karlsruhe, now part of the Karlsruhe Institute of Technology (KIT).

For many bio-inspired surface structures (Abbott and Gaskell 2007) a fabrication technique for the integration of nanostructures on three-dimensional surfaces is required. The integration of selective nanostructured surfaces in freely shaped three-dimensional cavities is a challenge in replication technology. Unlike to stochastic methods, the patterning by hot embossing or nanoimprint allows the structuring of well designed structures on surfaces. Especially in hot embossing the replication is typically limited to structures with vertical sidewalls allowing the demoulding of the replicated structures (Worgull 2009). Because of the precise, one directional demoulding path the replication of structures, even nanostructures, perpendicular to the demoulding direction will damage the structures. The strength of patterning by hot embossing is the possibility to create different kinds of surface patterns on one the same part. In particular, surface sub-μ structuring can be designed to introduce a structural gradient which can generate a driving force to move liquid samples along lotus like structured surfaces (Worgull et al. 2008a).

To take an advantage of the surface structures the integration into a more complex, often three-dimensional system is essential. Therefore technology of microthermoforming can be used. Microthermoforming of thin polymer films, with local surface modifications, was described by Giselbrecht et al. (2006). This approach of microscale thermoforming of thin polymer film substrates overlaid with a split local modification of the films is called

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‘SMART’, which stands for ‘substrate modification and replication by thermoforming’ (Truckenmüller et al. 2008). Geometrical modifications of nanoscale surface modifications due to uniaxial and biaxial stretching of the polymer film are measured, described, and compared with an analytical model by Heilig et al. (2010).

This paper presents first results of the improved process, based on a new thermoforming machine. Temperature stability and pressure distribution is highly improved by the newly designed control unit. Reproducible variations in pressure and temperature distribution can be archived at reduced cycle times. Thermoforming of high temperature polymers up to 350°C is possible.

2 Process and technology

The process to fabricate complex three-dimensional microstructures with multiscale features, e.g. nanostructured sidewalls, fuses on a combination of two replication processes: hot embossing and microthermoforming.

In a first step the polymer film has to be surface-structured. Replication of the surface structures (sub micron structures) is done by hot embossing (thermal nanoimprint). In a second step the surface-structured polymer film is formed into a complex three-dimensional shape by microthermoforming.

2.1 Replication of surface structures by hot embossing

In a first step the local surface modifications will be embossed onto the surface of a thin polymer film by hot embossing. Hot embossing, comparable with thermal nanoimprinting, is a microreplication technology where a melted polymer is embossed into a heated mould insert (Heckele and Schomburg 2004). There are several approaches to fabricate the mould insert. If a plane, periodic pattern of the surface modification is required, interference lithography is a good way to produce consistent nanostructures (Lu and Lipson 2009). Structure geometries may be sinusoidal, parabolic, lamellar or conical. The nanopattern is written into a positive or negative resist by two or three interfering laser beams. After development, the structures can be transferred into nickel by electroplating.

For surface modifications with variable patterns, electron beam lithography is a well-suited method. In this case, the nanopattern is written into the resist, for example polymethylmethacrylat, by a focused and well-guided electron beam. To fabricate the mould insert which is a thin metal plate consisting of the desired structures, the developed resist layer has to be transferred into nickel by electroplating.

The resist layer is coated with a 5–10 nm thick chrome layer by evaporation deposition. The chrome coating is needed as adhesive layer. Then a 20–30 nm thick gold layer is deposited on the chrome layer. The vapour deposited gold layer is needed as electrode in the electroplating process. On the gold layer nickel is deposited during electroplating up to a typical thickness of 500 µm. Finally the structured resist is separated from the electroplated nickel film. The thin nickel metal film is so called shim moulding tool.

The shim moulding tool is mounted on a metal substrate and is called mould insert. The mould insert is mounted on one side of a hot embossing machine as shown in Fig. 1. A metal plate with a rough surface, the so-called substrate plate, is positioned on the opposite side. The polymer film is positioned between the mould insert and the substrate plate.

After closing the vacuum chamber (not shown in Fig. 1) and evacuation of the enclosed volume, the thin polymeric film is heated above glass transition temperature for amorphous polymers or to the melting temperature for semi crystalline polymers and the embossing force is applied. Moulding force and temperature are the main parameters in this process stage. During force- and velocity controlled moulding, the cavities of the mould insert are filled with the viscous polymer melt. After filling the cavities, mould insert and substrate plate are cooled down. During cooling, the moulding force is maintained to compensate the shrinkage of the polymer. Demoulding fuses on a stronger adhesion of the residual layer on the substrate plate. The average of the measured demoulding stress is typically in a range of 0.2–0.5 N/mm^2. Demoulding temperature and the quality of the nanoimprinting tool are the major parameters responsible for successful demoulding (Worgull et al. 2008b and Kabanemi et al. 2009).

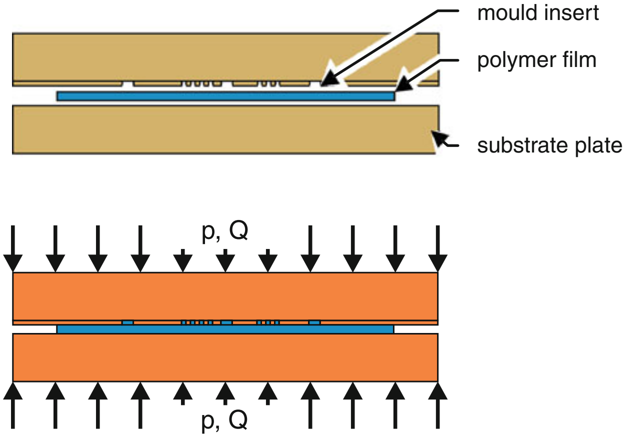


Fig. 1 Schematic view of the hot embossing process. Lateral dimensions of the structures are between 200 nm and 17 µm. The process is similar to thermal nanoimprinting

2.1.1 Implementation of hot embossing

Implementation of surface structuring by hot embossing, with examples of three different surfaces structures (27 μm, 500, 200 nm) and two different materials (PP and PMMA).

Cuboids with lateral dimensions of 17 × 17 μm and a height of 21 μm are hot embossed onto PMMA film (Degalan G7E 100 μm) as an example of larger surface-modifications. These cuboids structures with lateral dimensions of several microns are transferred into a resist (AZ 9260) by UV lithography from a chrome mask. Figure 2 shows the nickel mould insert after electroplating. Surface roughness is a result of the titanium oxide adhesion layer (5–10 nm) between the silicon wafer and the gold electrode (20–30 nm) for electroplating. The moulding temperature of the hot embossing process step was around 160°C at 20 kN moulding force. Figure 3 shows a detail of the structured area of 50 × 50 mm.

The replication of periodic nanostructures with lateral dimensions of 200 nm and a pitch around 400 nm was done in polypropylene (NOWOFOL PP-HM 100 μm) at 165°C and 40 kN moulding force. The size of the structured area is 19 × 19 mm. Figure 4 shows a SEM image of the mould insert. In Fig. 5 a SEM image of the hot embossed polypropylene film is shown.

A third type of sub micron structure was produced with silicon etching technologies. The silicon master structure was coated with a 5–10 nm thick chrome layer by evaporation deposition. The chrome coating is needed as adhesive layer. Then a 20–30 nm thick gold layer is deposited on the chrome layer. The vapour deposited gold layer is needed as electrode in the electroplating process.

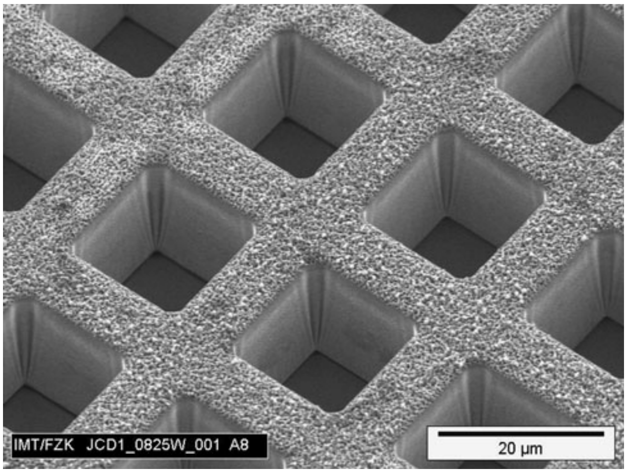


Fig. 2 Nickel mould insert with cuboid holes. Lateral dimensions of holes are 17 × 17 μm, with a depth around 21 μm. Width of bars is 8 μm. The structure is written into a resist (AZ 9260) by UV lithography from a chrome mask and transferred into the nickel mould insert by electroplating

Figure 6 shows the electroplated nickel insert with cubic structures of 500 nm. This structure was also replicated into PMMA film (Degalan G7E 100 μm) at temperatures around 135°C and 30 kN moulding force (Fig. 7). The size of structured area is 10 mm in diameter.

2.2 Process and technology of microthermoforming

The major process steps of microthermoforming are shown in Fig. 8. The surface structured polymer film is placed into the thermoforming machine between the microstructured mould and the counter plate. The chamber is evacuated and

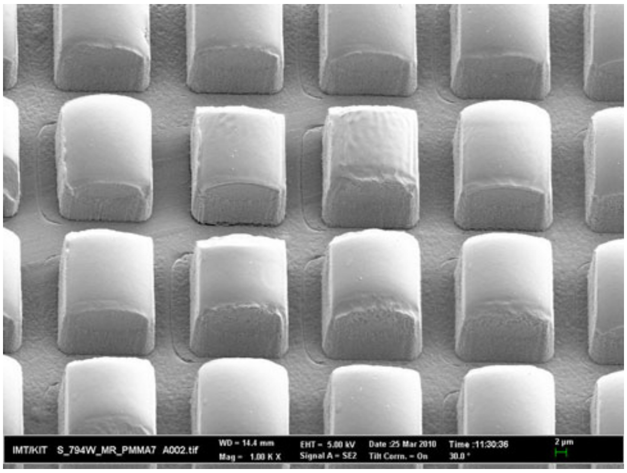


Fig. 3 Replication of cuboid holes in PMMA (Degalan G7E 100 μm). Lateral dimensions of pins are 17 × 17 μm, with a height around 21 μm. Replication was done with hot embossing at around 160°C and 20 kN moulding force. Size of the structured area is 50 × 50 mm

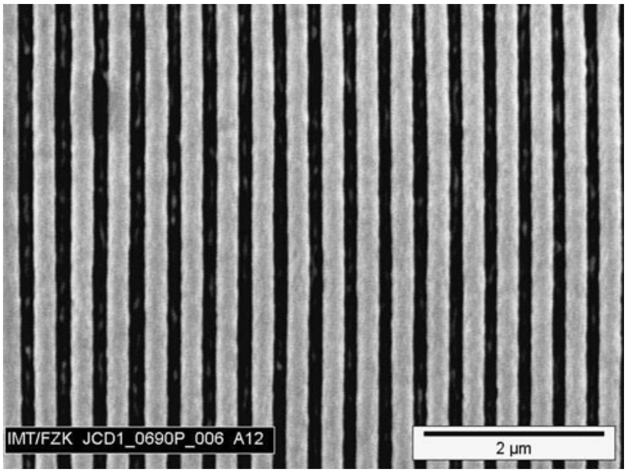


Fig. 4 Nickel mould insert with line and space structures. Lateral dimensions are 200 nm with a pitch around 400 nm. The line and space structure is written into a resist by interference lithography and transferred into the nickel mould insert by electroplating

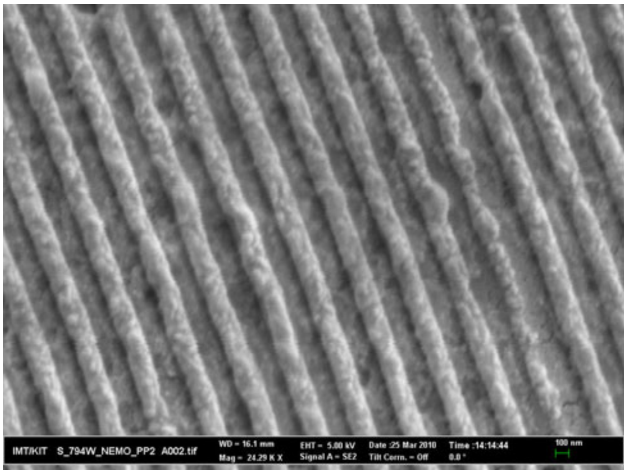


Fig. 5 Replication of line and space structures in polypropylene (PP-HM 100 µm). Replication was done with hot embossing at around 165°C and 40 kN moulding force. Size of the structured area is 19 × 19 mm

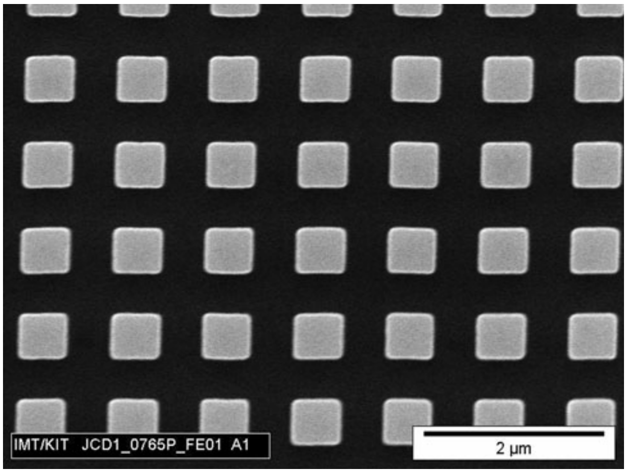


Fig. 6 Nickel mould insert with cubic pins. Lateral dimensions of pins are 500 × 500 nm, with a height of 500 nm. Space between pins is 500 nm. The structure is produced with silicon etching technology and transferred into the nickel mould insert by electroplating

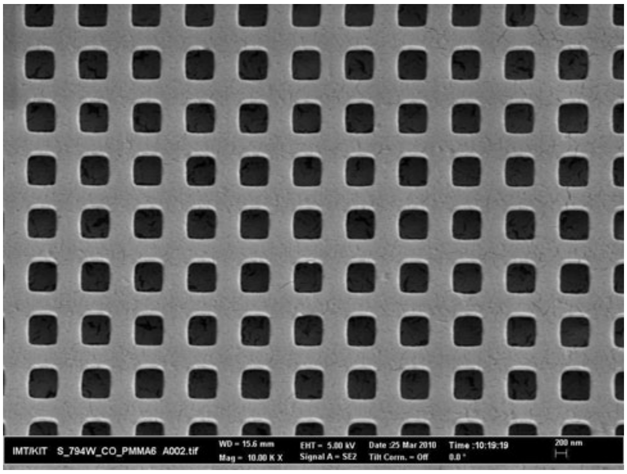


Fig. 7 Replication of cubic pins in PMMA (Degalan G7E 100 µm). Lateral dimensions of holes are 500 × 500 nm, with a height around 500 nm. Width of bars is 500 nm. Replication was done with hot embossing at around 135°C and 30 kN moulding force. Size of the structured area is 10 mm in diameter

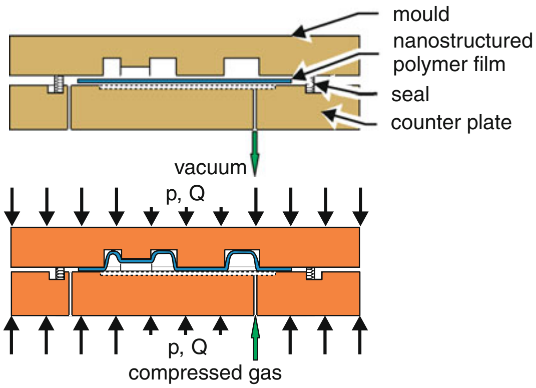


Fig. 8 Schematic view of the micro thermoforming process. A thin surface structured polymer film is clamped and heated up in the range of glass transition temperature and will finally be formed by gas pressure into microcavities

closed completely to achieve thermal contact between the machine plates and the polymer film for heating. Heating of thin surface structured polymer films (<100 µm) cannot be done outside of the thermoforming machine (pre heating) because of the low heat capacity and the risk of destroying the surface modifications.

By heating the mould and the counter plate, the polymer film is heated close to glass transition temperature (105–115°C for PMMA and 155–165°C for PP) but remains in a rubber-elastic state. In this temperature range the internal polymeric structure of the polymer film and the surface structure are preserved. To mould the surface structured polymer film into the micro cavities of the moulding tool, pressurized gas with a pressure in the range between 0.5 and 5 MPa is applied, while the polymer film is clamped to its surroundings. The polymeric film is formed by the gas pressure into the evacuated micro cavities of the mould. Because of the different temperature ranges of hot embossing and thermoforming the surface modifications will not be damaged while their carrier layer, the polymer film, will be formed into the microcavities of the mould. The cohesion of the polymer film is maintained. Because of the difference between the height of the nanostructures and the thickness of the carrier layer the forming process is determined by forming of the carrier layer. Stretching of the carrier layer corresponds also to the kind of geometry of the mould insert. Here uniaxial or biaxial strain can occur which determine the distribution of the surface modification in the final shape.

The microthermoformed polymer film will be cooled down to demoulding temperature under the load of the gas pressure. After achieving the demoulding temperature, typically below glass transition temperature of the polymer, the gas pressure is set to ambient pressure and the microthermoformed polymer film can be demoulded manually.

The mould for microthermoforming is typically made of brass or steel (Fig. 9). Polymeric moulds can be used for prototyping or as carrier layer of the microthermoformend parts (Heilig et al. 2010). Therefore a mould of the same material as the surface structured polymer film is used. It can be structured by hot embossing, but any other structuring method can be used as well. Because of the same material of surface structured polymer film and polymer mould, bonding between the polymer film and the mould can be achieved during microthermoforming.

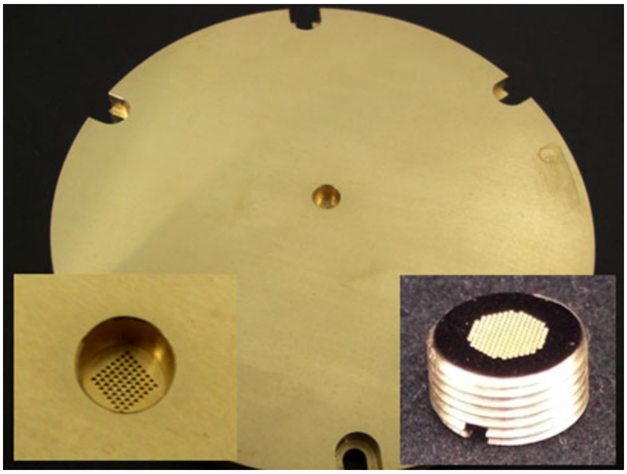


Fig. 9 Mould for multiscale microthermoforming made of brass. Cavities of the interchangeable insert are produced by micro milling. Diameter of insert is 5 mm with 300 μm diameter of the milled structures

2.3 Process parameters of microthermoforming

The behaviour of polymeric materials during thermoforming is strongly affected by the stress level, the rate of loading and the temperature (Argon 1973). Therefore, a precise control of the process parameters is necessary, especially during microthermoforming with the reduced heat capacity of thin polymer films (film thickness typically below 100 μm) and the small volume of the microcavities.

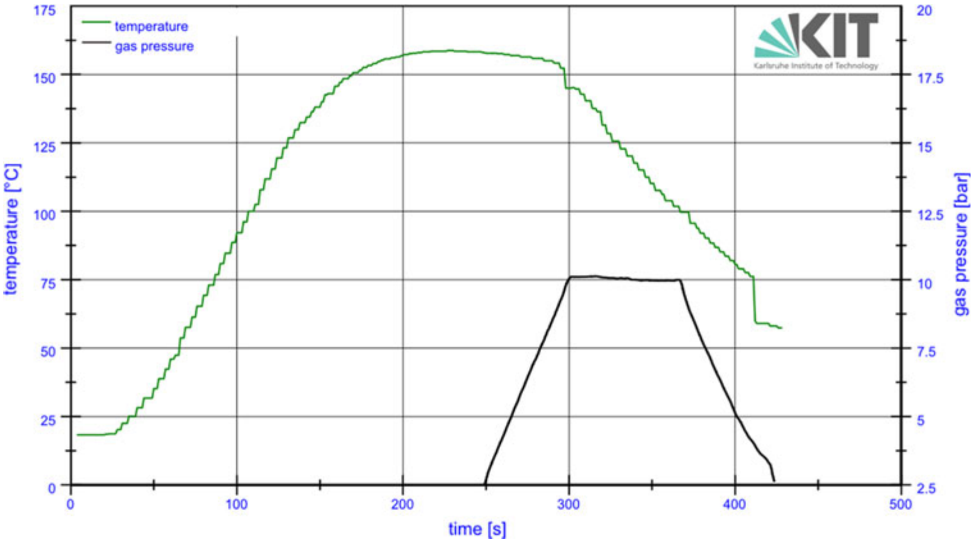
Microthermoforming has to be done in a small temperature range. The thermoplastic polymer has to be in a softened state, so that large polymer deformations are possible without breaking, nor melting the polymer (Sweeney et al. 2009). For amorphous polymers this temperature range is just above the glass transition temperature. Semi crystalline polymers are typically formed just below melting temperature.

Applied pressure level and rate of pressure rise are the other two technical main parameters. Makradi et al. (2008) showed that the mechanical characteristics of the final products, which are mainly controlled by the final polymer film thickness and orientation of the polymer molecular chains, depend strongly on the process temperature and the applied pressure profile.

2.3.1 Control of process temperatures

For the forming process the polymer film is heated to a softened state. The low heat capacity of thin polymer films requires that the mould and counter plate (Fig. 8) is also heated and hold in the forming temperature range until the full gas pressure is applied. To get better response during heating and reduce cycle times, mass of heated parts (mould, counter plate and heating plates) is reduced and thermally decoupled from the cooling device. After the full

Fig. 10 Temperature and pressure profile during microthermoforming of polypropylene (Nowofol HM). Temperature was set to 156°C and hold in a temperature range of ±2°C. Forming pressure was set to 10 bar (1 MPa). A cycle time of 5 min is accomplished (75–156°C and back to 75°C)



gas pressure is reached, cooling is started by coupling and activating the cooling device. Therefore the heating plates are pressed against water-cooled machine plates.

In Fig. 10 temperature and pressure profile during microthermoforming of polypropylene (Nowofol HM) is presented. Forming temperature was set to 156°C and was held in a temperature range of ±2°C until forming pressure is applied. Rate of temperature change was 65 K/min during heating and −40 K/min during cooling.

2.3.2 *Control of process pressure*

Microthermoforming requires precise control of the applied pressure profile. An automated forming pressure control unit, for presetting the pressure rise, level and fall was designed. Pressure rise and fall can be set between 10 and 40 kPa/s with a linearized pressure distribution profile. Maximal level of gas pressure can be up to 5 MPa and is hold constant by the controller at the preset value. The challenge was to achieve these parameters at the small (gas-) volumes of the microcavities, typically around 500 mm^3.

As shown in Fig. 10, the gas pressure of 1 MPa was applied and hold in a range of ±30 kPa for microthermoforming of polypropylene (Nowofol HM 100 μm). Pressure rise and fall was set to 20 kPa/s.

3 Results and conclusion

The integration of selective surface modifications in freely shaped three-dimensional cavities is expected to be a very

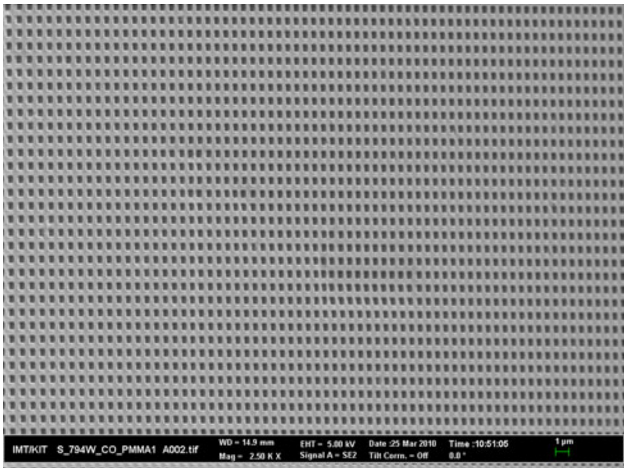


Fig. 11 Surface structure of Fig. 7 on PMMA (Degalan G7E) after microthermoforming. Image was taken on next to a microcavity of the microthermoformed part

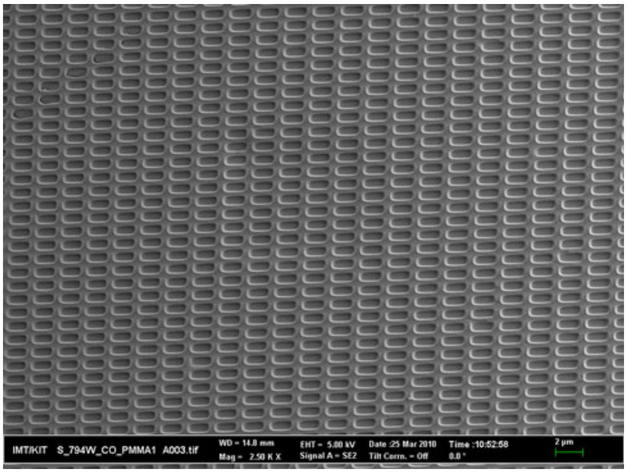


Fig. 12 Surface structure of Fig. 7 on PMMA (Degalan G7E) after microthermoforming. Image was taken in a microcavity of the microthermoformed part

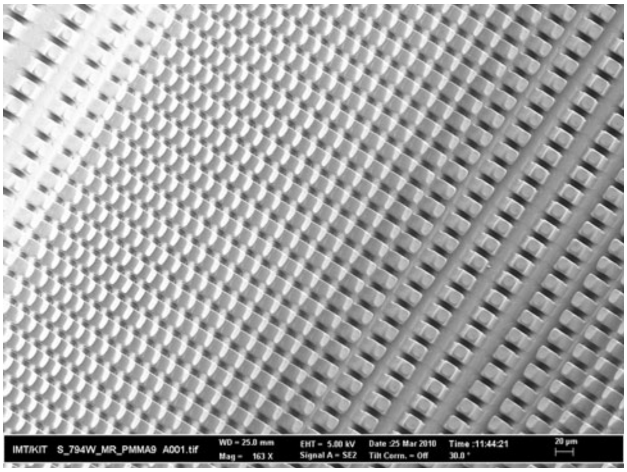


Fig. 13 Surface structure of Fig. 5 on PMMA (Degalan G7E) after microthermoforming. Image shows the top view on the microthermoformed part

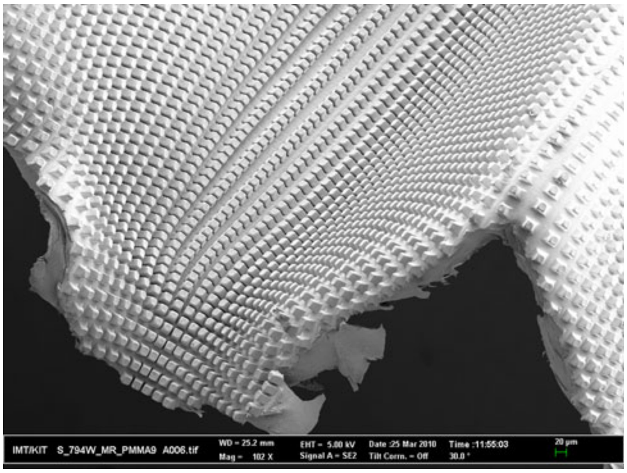


Fig. 14 Surface structure of Fig. 5 on PMMA (Degalan G7E) after microthermoforming. Image shows a tilt view (30°) on the microthermoformed part

useful technology e.g. for life science applications. It is demonstrated, that a combination of hot embossing and microthermoforming is a suitable approach to fulfill this task. With the enhanced temperature and forming pressure control unit, it is possible to take advanced control over the microthermoforming process. It is possible to analyse the process of microthermoforming in detail and compare it with analytical calculations and simulations. The influence of microthermoforming on the surface structure of the polymer film is illustrated in Figs. 11–14. The grid structure of Fig. 7 on PMMA (Degalan G7E) is stretched during microthermoforming. Figure 11 shows an area of the microthermoformed part where less to none stretching of the polymer film was applied. In Fig. 12 the surface structure is stretched in a factor of four in one direction. It is located on the ground of a micro channel. Due to the different order of magnitude, surface structure in the sub-micron domain, cavities of microthermoformed part in the micron to mm domain, it is not possible to illustrate both structure in one image.

A top view of the surface structure of Fig. 3 after microthermoforming is shown in Fig. 13. A detailed view under 30° tilt is shown in Fig. 14.

With the described combination of hot embossing and microthermoforming it is possible to replicate microparts with multiscale features. Outer dimension in the macroscopic length scale and functional design parts in the microscopic length scale can be combined with nanoscopic structures in an affordable technology. Figure 15 shows a picture of a multiscale cell cultivation container with outer dimensions of 6 mm in diameter and 5 mm in depth with cups of 300 µm in diameter and depth on its ground. Figure 16 illustrates the surface structure with the optical refraction due to the nanoscale structuring with an intermittency of 400 nm of the surface structure (Fig. 5).

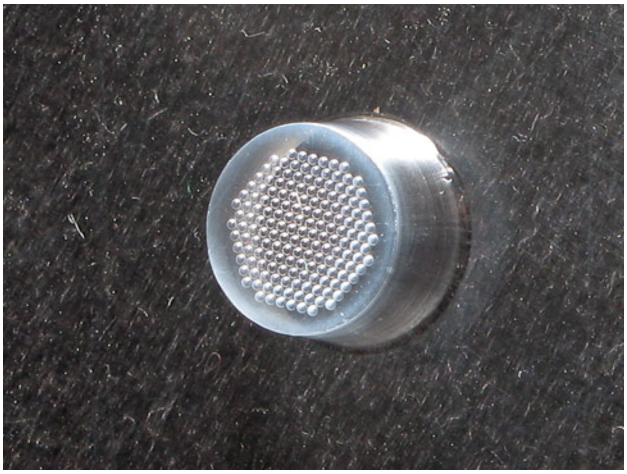


Fig. 15 Backside view of multiscale container for cell cultivation, produced by Microthermoforming with mould of Fig. 9. Outer dimensions of 6 mm in diameter and 5 mm in depth with cups of 300 µm in diameter and depth on its ground (top in picture)

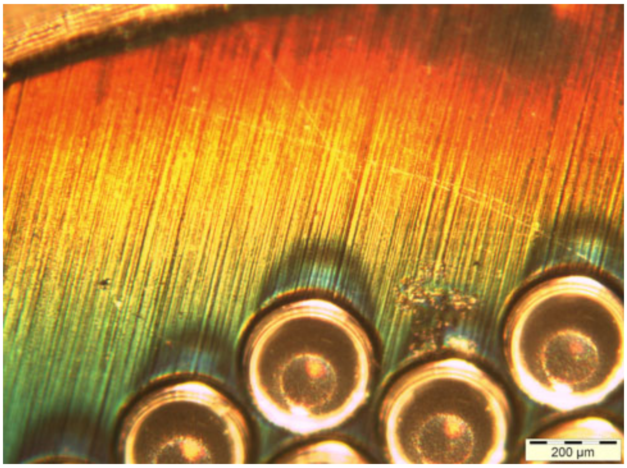


Fig. 16 Optical refraction on the cell container (Fig. 15) due to the nanoscale surface structuring of Fig. 5 with an intermittency of 400 nm

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