

Hot embossing of micro and sub-micro structured inserts for polymer replication

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Abstract Today replication of microstructured parts is state of the art in laboratory and commercial use. Beside the process of injection molding hot embossing enables the accurate replication of polymer structures in a broad variety of thermoplastic polymers even in the nanometer range. Characteristic for the most replication processes dealing with thermoplastic polymers is the use of microstructured mold inserts based on metals. In this paper we describe an alternative to the established mold inserts—the use of so called interstage mold inserts. These interstage mold inserts are replicated in high performance polymers and technical thermoplastics and can be fabricated many times by a previous replication step from a master even in the sub-micro range. Aspects like suitable material combinations, demolding behaviour, long time stability, production rate, and the quality of structures will be discussed. Because of the high flexibility the process of hot embossing is used for the fabrication of the microstructured interstage mold inserts and their replications.

1 Introduction

Polymer parts with functional structures in the micro- and sub-micrometer ranges entered the consumer mass market long ago. However, research and development of new micro- and sub-micro-structured products like for lab-on-chip applications or optical devices are still cost-intensive and time-consuming. Therefore the fabrication of polymer microstructures by replication of metal masters is developed

to a common technology for research and industry in the last decade (Kato et al. 2008).

During large serial production the lifetime of mold inserts is an essential aspect for the costs in commercial applications. During development of systems and in laboratory use the lifetime of a mold is only partially an issue. Items like flexibility and quick change of some features of the mold is in the foreground of interest. An opportunity to implement these requirements is the use of so-called interstage mold inserts, replicated by an already existing metal master. The advantage is that modifications of the design can be made at these interstage mold inserts. Using this combination the expensive master can be used as a basis for further modifications. The challenge is to replicate the so-called interstage mold inserts in thermoplastic polymer material allowing the use as a mold insert for another thermoplastic material. Process conditions and material combinations will be investigated by the replication process of hot embossing. The lifetime of the interstage mold inserts will be determined also like the quality of the replicated parts compare to the master and the interstage mold insert.

2 Hot embossing technology

The process of hot embossing is besides micro injection molding and UV-nanoimprint processes one of the established processes for the replication of micro and nano-structures. Especially in laboratory use and for low volume production hot embossing shows a number of advantages, in particular the high grade of flexibility regarding process modifications. Because of the short flow paths and the quick change of polymer, the open tool technique hot embossing fulfils the requirements of high-precision

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polymer replication using a wide range of thermoplastics. Structure sizes in the macro range down to the nanometer range can be replicated on areas up to 10 inch diameter.

The common alternative replication process micro injection molding, is particularly suitable for high-volume production and has become indispensable for industrial polymer replication. Micro injection molding is the most popular polymer microreplication technology due to short cycle times, low costs of high-volume production, and a broad variety of processable thermoplastic polymers (Piotter et al. 2002). Compared to UV nanoimprinting or hot embossing, however, this replication technology is still limited when replicating thin, flat parts with high aspect ratios in the lower micro- and nanometer range. The fast injection and cooling of the polymer melt, together with long flow distances, result in higher inner stresses which adversely affect microoptical systems like photonic crystals or microfluidic components and a subsequent treatment by bonding, for instance, becomes impossible. UV nanoimprinting is suited for the production of high-precision polymer structures in the nanometer range with high aspect ratios, but is limited to polymers which are UV curable. For large-area parts with structures of different orders of magnitude, this process still is under development (Heckele and Schomburg 2004).

Hot embossing allows for the replication of all known thermoplastic polymers especially of large-area microstructured parts with low inner stress, when using semi-finished products like cell casted polymer sheets. Due to the localized direct polymerization of the polymer foil in a frame with the adjusted thickness the polymer is not subjected to thermal or mechanical treatment. During this

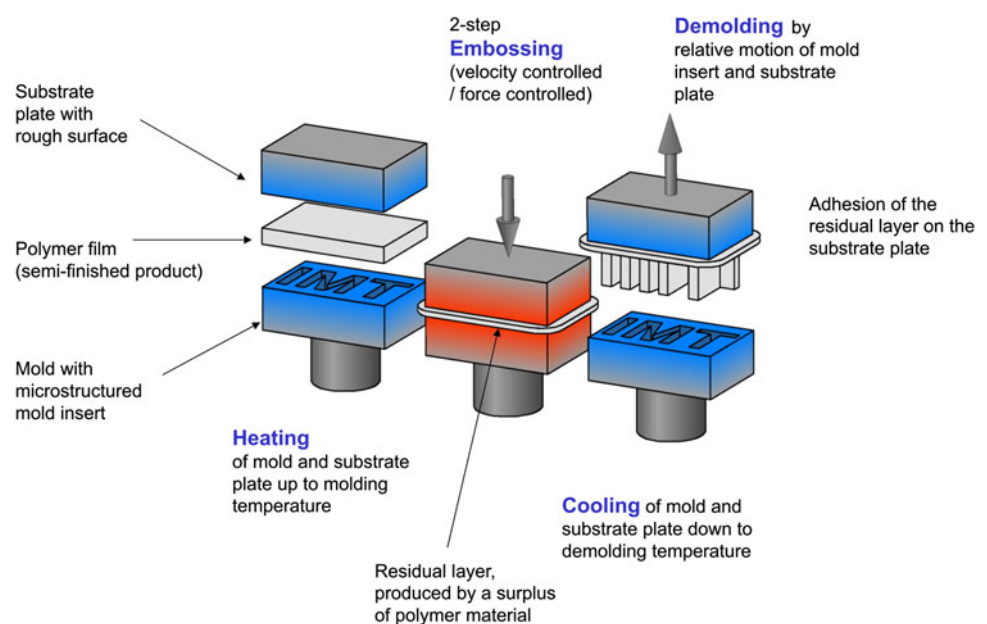
production it does not lead to any inner stress formation of the polymer foil. When using foils as a raw material, flow distance of the polymer melt into the microstructured cavities is very short and does not induce any additional material stress.

The increase in demand for polymer parts with microstructured features has also resulted in increasing requirements on microstructured parts. Microreplication technologies like micro injection molding, hot embossing, or UV nanoimprinting can be tailored to meeting these requirements.

Hot embossing is based on heating up a polymer raw material until it softens and molding it into a metal insert, the master structure. The raw material consists of a polymer foil with a typical thickness from below 100 μm up to several millimeters. After placing the foil into the hot embossing machine, the vacuum chamber has to be closed and evacuated to prevent inclusions in the cavities between the master structure and polymer foil. The most time-consuming step of hot embossing is the heating of the insert, foil, and substrate plate to above the glass transition temperature in case of amorphous polymers or the melting temperature in case of semi-crystalline polymers. By this step, the polymer foils is rendered malleable for the forming step. Figure 1 shows a typical hot embossing process (Worgull 2009).

Forming is performed by a velocity- and force-controlled embossing step with velocities between one to tens of micrometers per second and an embossing pressure between 10 and 100 MPa, which is controlled by a high-precision load cell. To ensure homogeneous temperature and pressure distribution during the replication process, the

Fig. 1 Schematic representation of a typical hot embossing process



force and temperature are kept constant over a defined time, depending on geometries, structure size, and flow ranges. The hot embossing machine consists of precise vertical guidance units and highly parallel traverses comparable to a universal testing machine. Using this embossing machine, flat and parallel polymer parts can be replicated.

After filling of the cavities and structures, the polymer part is cooled down under constant embossing force to prevent shrinkage. The demolding temperature has to be even more accurate than the embossing temperature, because demolding is the crucial process step of hot embossing. During cooling of the polymer melt in the insert cavities, the metal tool and the polymer part shrink differently, which results in forces of the polymer to the sidewall of the structure as shown in Fig. 2.

These forces may damage the replicated structure. Consequently, precise control of temperature, force, and position during demolding is essential for high-quality replication. During the process, demolding is performed automatically due to the higher stiction of the polymer part on the rough substrate plate. The roughness has to be adapted to the insert structure to overcome stiction on the insert when opening the machine. Demolding temperature typically is a few degrees below the glass transition temperature or the melting temperature and the demolding velocity is half of the embossing velocity.

Hot embossing even allows for a replication in the nanometer range with very low inner stress. Polymer replication by hot embossing is limited by the quality of the structure and surface of the metal inserts. Even nanometer roughness will be copied by the replication process. Side wall roughness of the insert will result in a corresponding roughness of the side walls of the polymer parts due to vertical demolding.

Due to precise temperature and force control a broad variety of polymers can be used for replication by hot embossing. The machine can be modified for replication of thermoplastic polymers above the glass transition temperature or crystalline melting point of 350°C. Hot embossing is especially suited for producing delicate microstructures with high aspect ratios and low tension on thin layers. Production quantities typically range from a single copy up

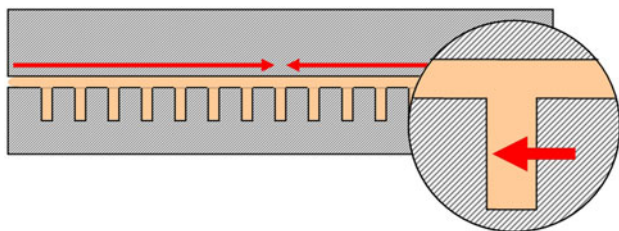


Fig. 2 Schematic depiction of the origin of demolding forces

to thousand parts. Higher volumes can be produced more efficiently by micro injection molding (Heckeles et al. 1998).

The mold insert and setup can be changed within a few minutes by fixing substrate and tool plates. The simple geometry of the tool plate decreases the production costs of tools significantly compared to micro injection molding, which makes hot embossing an economically efficient process for the production of up to several hundreds of parts.

The high quality of the inserts required for accurate replication leads to improved fabrication technologies and results in high costs of the insert compared to material costs and costs of replicated parts. In micro injection molding, the high quantity of replicated parts due to an automated process and short cycle times compensates high tool costs. As hot embossing is usually applied to produce only tens to hundreds of parts with cycle times between a few minutes to half an hour, costs of the tools are important to production and replication is comparatively time-consuming. By using polymer interstage mold inserts for hot embossing, fabrication costs of the tools can be decreased due to the smaller area of the metal tool and parallel production with several polymer interstage mold inserts can significantly reduce the production time. Reduction of wear of the inserts may improve the replication efficiency. Use of polymer inserts for further replication increases the service life of expensive inserts and, thus, allows for the economic production of polymer micro parts by hot embossing to several thousands of parts.

3 Polymer inserts

3.1 Requirements on polymer inserts

The dimension of functional structures of replicated polymer parts is established in the lower micrometer and the sub-micrometer ranges. The smaller the structures of the polymer parts are, the more challenging is the replication due to an increasing ratio between the surface roughness and the structure size of the replicated part and, consequently, of the insert. These side walls of the insert need very accurate and smooth surfaces to minimize stiction. The quality of the insert and especially the surface of the side walls are decisive for a good replication. This leads to increasing requirements on insert fabrication. With increasing requirements, however, the costs of the insert increase as well and often, only one insert will be produced. By hot embossing, an expensive master insert can be replicated into suitable high-temperature polymers and this copy can be used for further replications. Costs for further inserts can be saved.

Hot embossed polymer inserts can also be used in parallel by integrating them in a large-area hot embossing machine. This improves the throughput especially of microstructures. In our experiments the hot embossing area was enlarged to 40,000 mm². The machines used were a Wickert WMP1000 with improved cycle times and embossing forces of up to 1,000 kN and a commercial Hex03 from Jenoptik Mikrotechnik shown in Fig. 3. For later stability investigations, 15 parts were embossed in parallel per cycle.

Use of hot embossed polymer inserts allows for a simple integration of additional structures like marks or references during the first replication step. A better machine integration is possible by placing protecting layers between the inserts and the polymer. An overlaying grid can be used for clamping the interstage mold insert onto the tool plate for further replications.

For the replication of polymer interstage mold inserts, different nickel inserts with periodic structures between 500 nm and 20 μm in lateral direction and heights between 1 and 25 μm were used. The nickel inserts were produced by electron beam writing, silicon technology, or multi-level etching for master production, followed by electroplating. The structured area was between 1,000 and 2,500 mm².

Usability of polymer inserts for further replications depends on the thermal and physical properties of the polymers. The main factor is the thermal stability of the polymer insert at the embossing temperature of the replication material. Therefore a gap of at least 20 K between the glass transition range, or the melting range respectively, of the insert polymer and the replicated polymers is decisive. Some polymers of the replicated part exhibited higher adhesion to a polymer insert, which partly hampered demolding, due to lower difference of thermal expansion coefficients of polymer–polymer combinations compared to polymer–metal, however, demolding forces may be decreased by using polymer inserts. Using polymer interstage mold inserts, the demolding temperature is typically

chosen to be a few degrees below that of the metal inserts, which guarantees solidification of the replicated polymer without the risk of high stiction to the insert side walls.

3.2 Interstage mold inserts replicated in high-performance polymers

For the first tests, high-temperature polymers with a softening temperature above 200°C were used. The most stable and accurate replication was reached for high-performance semi-crystalline polymers. Interstage mold inserts of liquid crystalline polymer (LCP CT-×100, Kuraray) and polyetheretherketone (PEEK Aktiv 1000, Victrex) showed no significant deformations after 50 replications in poly(methyl methacrylate) (PMMA Hesaglas VOS, Notz Plastics). The large difference between the replication temperatures of LCP and PEEK and technical thermoplastics like PMMA and PC allows for a huge number of replications without disruptive deformations (Griffiths et al. 2009). The difficulty in using high-temperature polymers for replication is the precise process control and high heating temperature for the replication of the interstage mold inserts in PEEK and LCP. Embossing temperatures between 327°C for LCP and 334°C for PEEK and embossing forces between 80 kN and 110 kN are required.

As most hot embossing machines operate at temperatures below 300°C, additional experiments were performed at processing temperatures below 300°C. Tetrafluoroethylene/hexafluoropropylene (FEP Symalit 1000, Quadrant EPP) showed good replication results at 260°C, but could not be used for further replication processes due to the insufficient hardness of the microstructure even at low temperatures. Hot embossed parts produced using an FEP interstage mold insert showed elongations of the structures of more than 150 % in the direction of polymer flow.

As another material for interstage mold inserts, polytetrafluoroethylene (PTFE Teflon, DuPont) was used at processing temperatures below 300°C. Although the melting temperature of PTFE is above 320°C, processing

Fig. 3 Hot embossing machines Hex03 (*left*) and Wickert WMP1000 (*right*)



of PTFE at temperatures between 270 and 280°C by a modified embossing process with embossing forces of 950 kN leads to best results and accurate replications. On the surface of replicated PTFE parts fibres with diameter below 1 µm occurred due to the fabrication of the PTFE foils. Figure 4 shows a PTFE interstage mold insert after one replication in polystyrene (PS Osstyrol SB, Hagedorn plastics).

For interstage mold insert replication, amorphous technical thermoplastics with glass transition temperatures below 140°C were used. Good replication results were reached for both PMMA and PS. Figure 5 shows the replication of a PS part out of a PTFE interstage mold insert at 135°C embossing temperature and an embossing force of 75 kN. Several PTFE fibers from the interstage mold insert have been transferred to the surface of the PS structure. This led to an inhomogeneous Teflon® coating on top of the PS part.

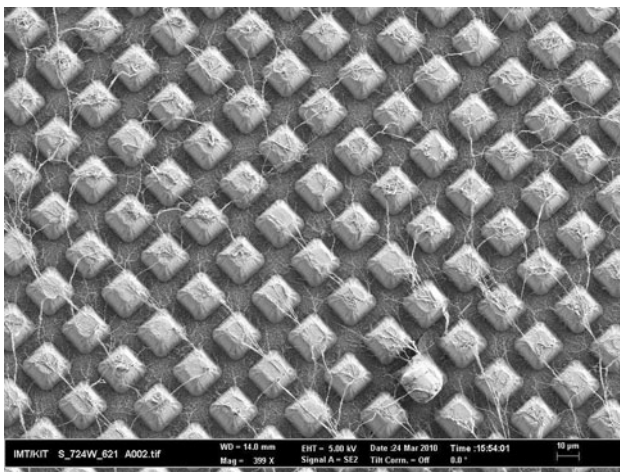


Fig. 4 SEM of hot embossed PTFE (Teflon, DuPont) cuboids with an edge length of 17 µm and height of 22 µm after one replication in PS (Osstyrol, Hagedorn)

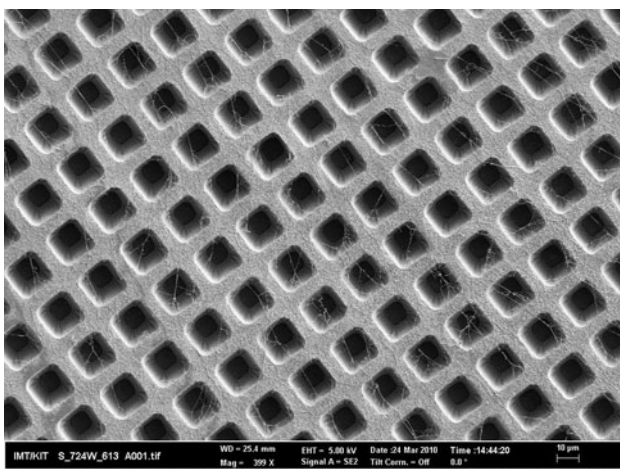


Fig. 5 SEM of a replicated PS part from PTFE interstage mold insert

3.3 Interstage mold inserts replicated in technical thermoplastics

Commercial standard hot embossing machines are operated at hot embossing temperatures below 220°C. For the use of interstage polymer inserts in commercial machines, further experiments were performed with technical thermoplastics. To fully utilise the available temperature range, polysulfone (PSU Lite U, Lipp-Terler) was used to hot emboss polymer inserts at a processing temperature of 220°C and an embossing force of 150 kN. The PSU interstage mold inserts were then examined for stability as described below.

But even common technical thermoplastics can be used for replication of standard polymers with glass transition temperatures below 120°C. Polycarbonate (PC Makrolon 2405, Bayer MaterialScience) was applied for the first replication due to its comparably high softening temperature. Further replication of this interstage mold insert, however, produced different results depending on the chosen material combination.

For the experiments with polycarbonate, a nickel insert with sub-µm structures was used. In the nickel insert, the cuboids were convex structures. This led to cuboid holes in the replicated PC insert. The distance between the cuboids was 500 nm in each direction and the height of the structure was 1 µm.

Several parts replicated in all materials, were measured by AFM. When using styrene acrylonitrile (SAN NAS 30, Ineos nova) for the replication of the PC interstage mold insert, hollow SAN structures occurred because of incomplete filling of the insert. Polyethylene (PE GUR 4120, Ticona) and polystyrene (PS) replications did not result in equally high cuboids, but in elongated thinner structures due to pinches during the demolding step with significant deformations of the cuboid geometry. In both cases, several cuboids were sheered out of the part and remained in the insert. Figure 6 shows an AFM measurement of a PS replication of a PC interstage mold insert. Figure 7 shows the corresponding SEM image. The height of the structures was elongated by more than 200 nm compared to the PC insert and the side wall showed clear pitches.

Good replication results were obtained by the combination of a PC interstage mold insert with polyvinyl chloride (PVC GH49, Rottolin) and PMMA. Figure 8 shows the interstage mold insert of PC with cuboid structures of 500 nm in edge length and 1 µm in height after one replication in PVC. The corresponding part embossed in PVC is shown in Fig. 9.

For the use of technical thermoplastics, it is of primary importance to choose the appropriate polymer combination to guarantee improvement of production and reduction of costs.

Fig. 6 AFM measurement of PS microstructures embossed using a PC interstage mold insert

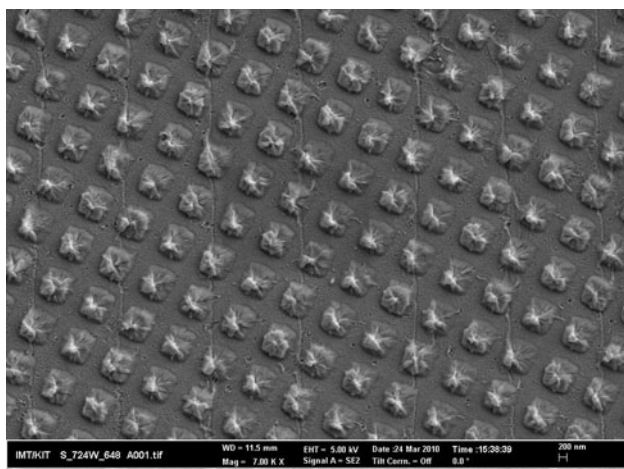
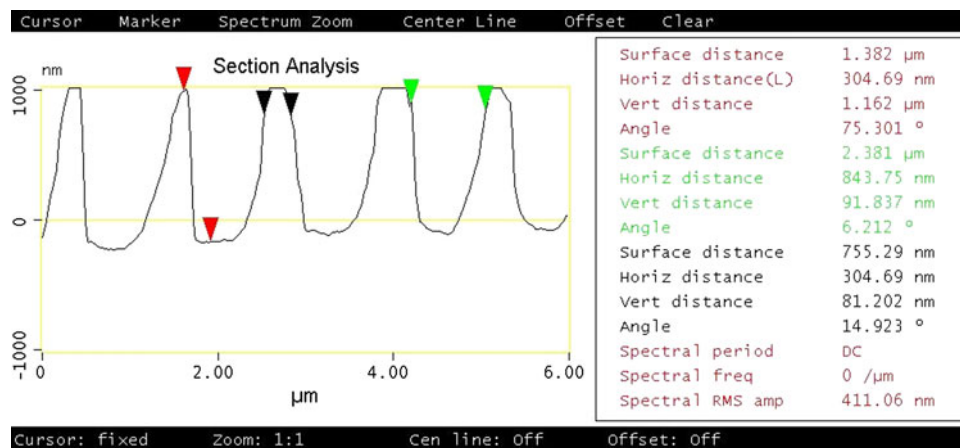


Fig. 7 SEM of PS microstructures embossed using a PC interstage mold insert

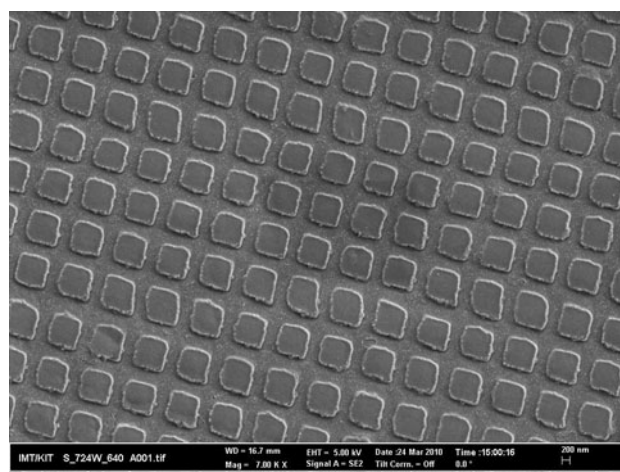


Fig. 9 SEM of the replicated part in PVC when using an interstage mold insert of PC

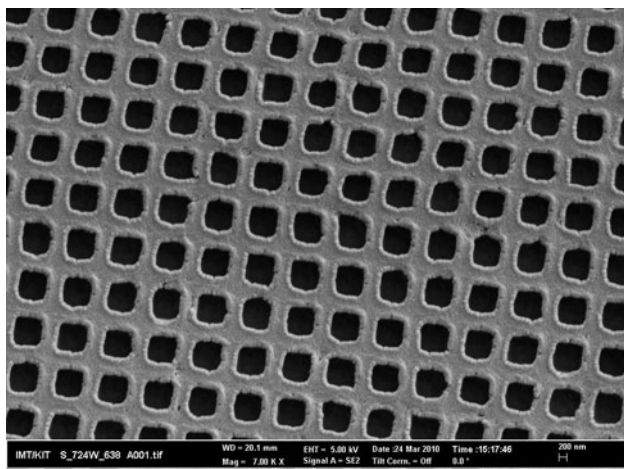


Fig. 8 SEM of a PC interstage mold insert with sub-μm structures after one replication in PVC. The edge length is 500 nm and the height is 1 μm

4 Stability and lifetime of embossed polymer inserts

Polymer interstage mold inserts were found to be generally applicable for further replications. Their applicability, however, is also dependent on the stability of the polymer insert during the subsequent replication processes. To study interstage mold inserts made of technical thermoplastics, the height of PC structures was measured by AFM before and after replication. The master insert consisted of cuboids with an edge length of 500 nm and a height of 1 μm. The height of the insert structures after replications in PVC, PMMA, and PS varied by 70 nm, which is comparable to the variations of material and process parameters during the replications. Figure 10 shows a cross section of a PC interstage mold insert before embossing, whereas Fig. 11 presents the measurements after one replication cycle. The height of the cuboids remains in the range between 940 nm and 1,010 nm.

Fig. 10 AFM measurements of a PC interstage mold insert before replication

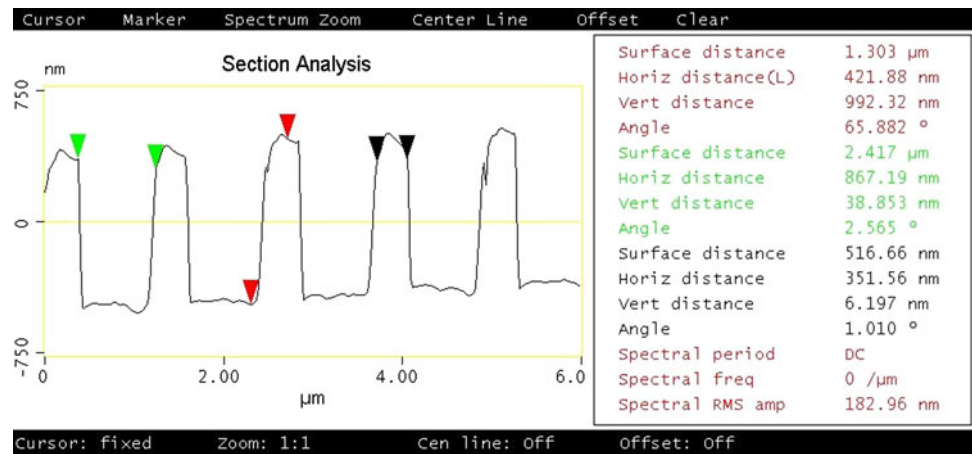
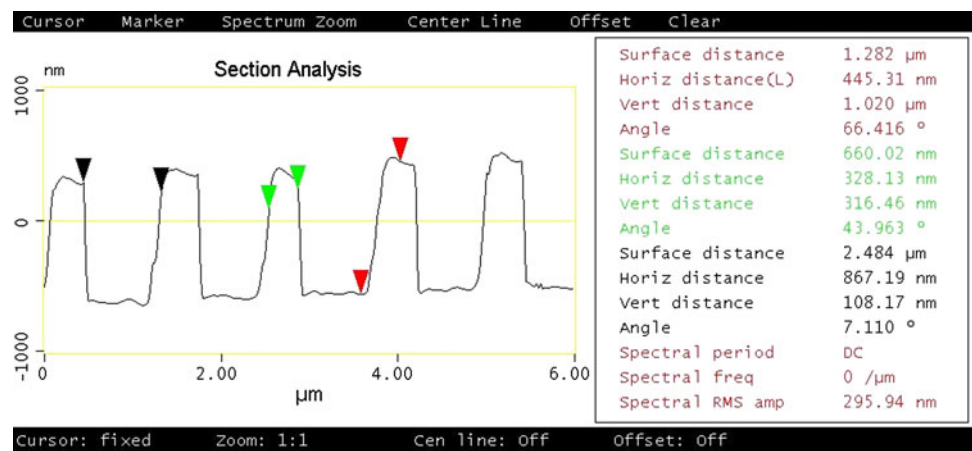


Fig. 11 AFM measurements of a PC interstage mold insert after one replication in PMMA



The stability of PSU interstage mold inserts was studied systematically for multi-level etched optical structures. For a fast and simple inspection of the embossed parts, a nickel insert with multi-level etched structures for optical diffraction was used. The replicated polymer parts were used as refractive optical systems. The patterns and pictures obtained revealed constructive and destructive interferences of coherent monochromatic light. The functionality of the replications was checked by using a laser beam pointing towards the replicated transparent part. The structure of the replicated part leads to interference behaviour and a picture comprises of high level regions out of the resulting interference. Each replicated part was inspected by examining the resulting interference picture (Waddie et al. 2006).

Only one nickel insert was used for all replications, but during the experiments, productivity was increased significantly by arranging up to 15 hot embossed polymer inserts in parallel on the available embossing area of 45,000 mm². Due to the large embossing area, the parts were replicated with forces of up to 800 kN to minimize shrinkage and guarantee the filling of all cavities (Worgull et al. 2008).

After ten replications each, the polymer inserts were measured by AFM to determine the height of the structure. Figures 12, 13, 14, 15 show the deviations of the structures during the replication processes. Significant deformations of the microstructures can be seen after 50 replications. Fillets over the hole embossing area and a loss of height of about 25 % were measured. The optical function, however, was sufficient even after 50 replications and showed the desired interference picture. After 10 and 20 replications, no systematic deformations were measured, the loss of the structure remained below 100 nm in height. This confirms the suitability of hot embossed polymer inserts for further replications.

5 Applications

Use of hot embossed interstage mold inserts may lead to new kinds of micro- and sub-micro geometries and products which cannot be replicated by conventional hot embossing technology. Due to the limitations of tool fabrication technology, some geometries are not replicable, but it is possible to fabricate the inverted structure. For

Fig. 12 AFM measurements of PSU interstage mold inserts before replications in PMMA

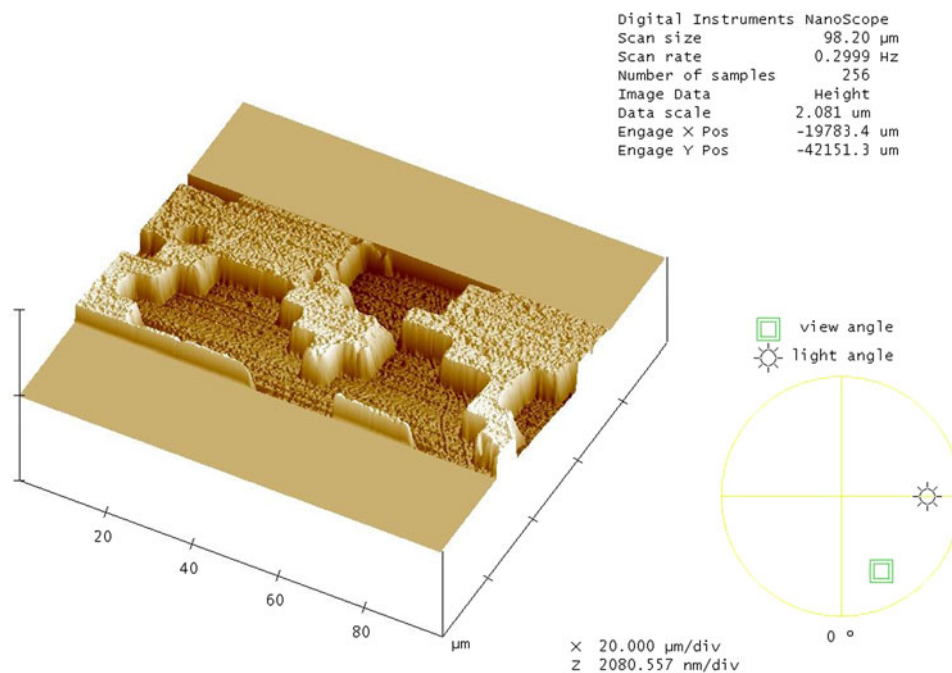
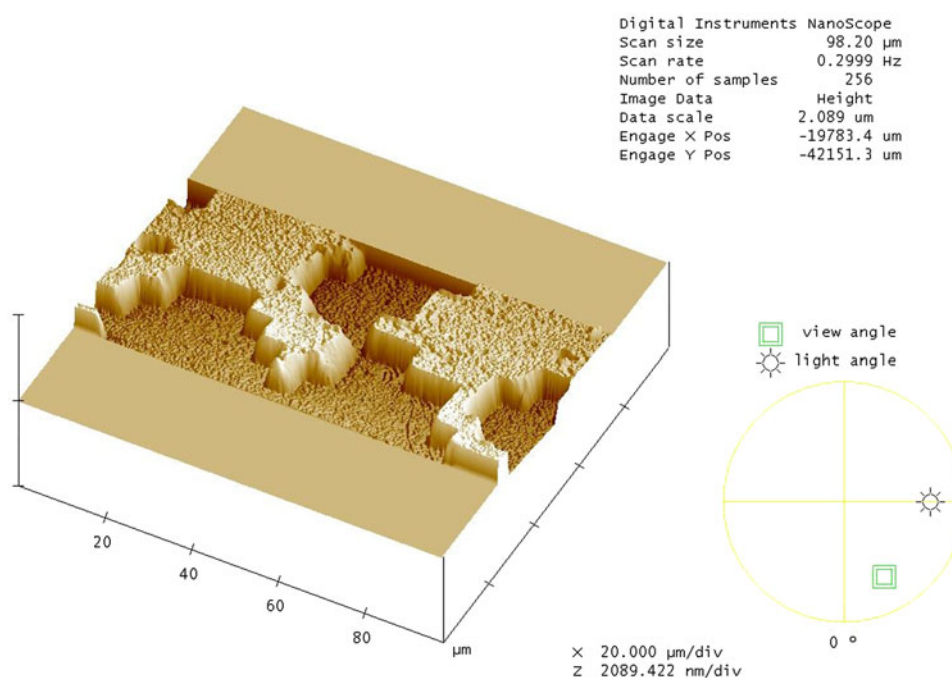


Fig. 13 AFM measurements of PSU interstage mold inserts after one replication in PMMA



example, metal inserts with free standing columns having a diameter in the lower micrometer range and aspect ratios of more than two are difficult to electroplate from a master structure due to the limited backfilling of deep vertical holes. Generation of the inverted structure is associated with less difficulties and results in holes in the metal inserts. The replication yields a polymer interstage mold insert with the desired insert geometry, which can be replicated in thermoplastic parts with an array of

microholes. A similar procedure is used for the replication of pyramidal holes. Protruding pyramids are milled directly with sharp edges. Recessed pyramidal holes require fabrication radii which influence the properties of the replicated polymer system. The inverse copy of the metal insert, however, shows these structures and allows for the replication of new polymer parts that could not be replicated before without additional processing steps (Wissmann et al. 2008).

Fig. 14 AFM measurements of PSU interstage mold inserts after 20 replications in PMMA

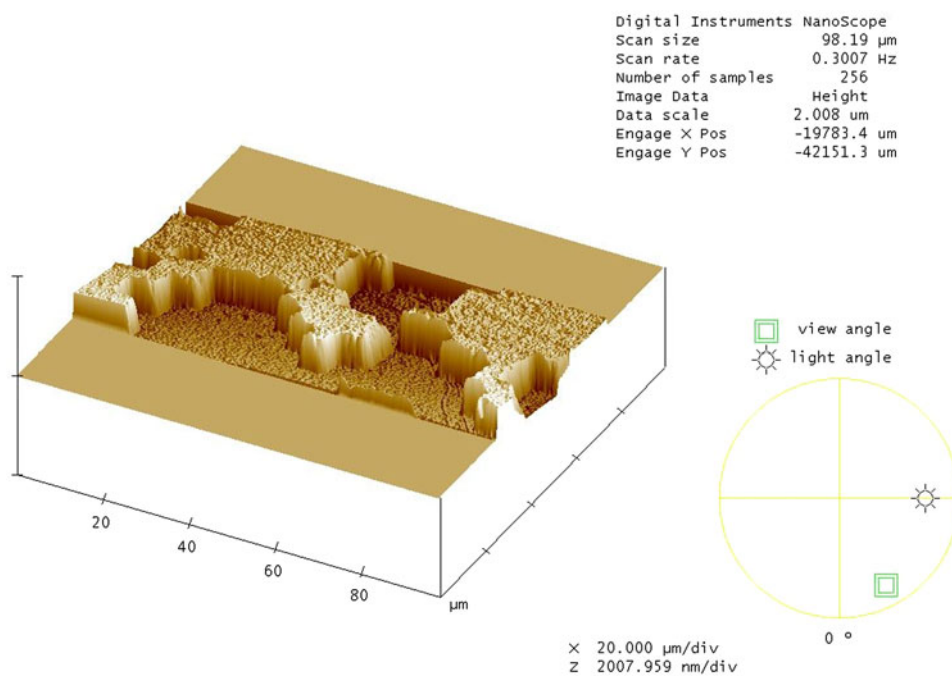
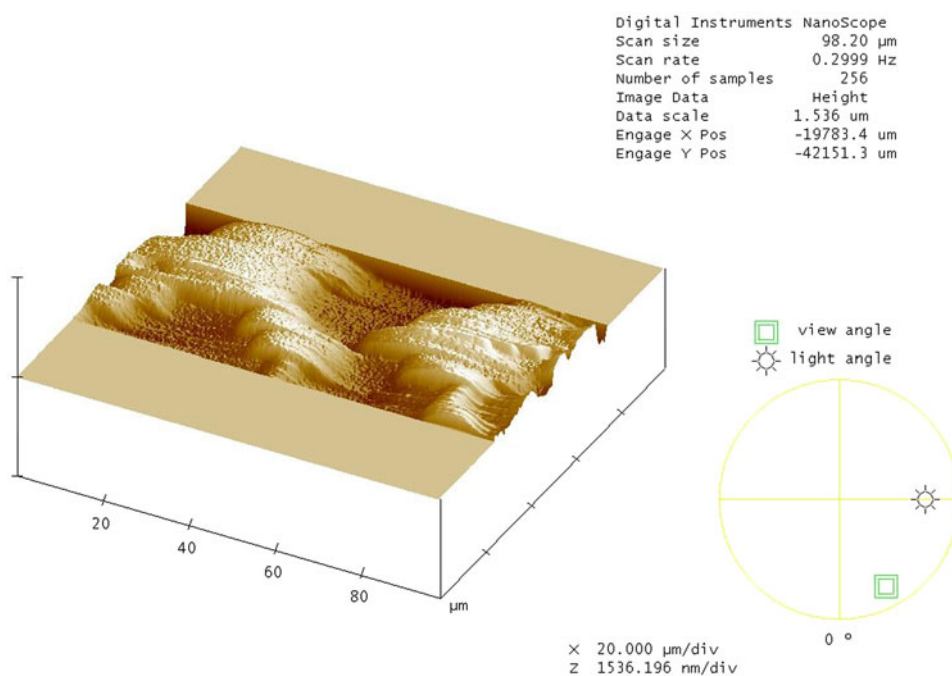


Fig. 15 AFM measurements of PSU interstage mold inserts after 50 replications in PMMA



The technique of hot embossed interstage mold inserts cannot only be used for the additional replication step, it is also suited for specific and located surface modification within microstructured polymer systems. For instance, 100 μm thick PSU foil was embossed with functional microstructures and used as disposable interstage mold insert. The microstructured foil, however, fixed to the tool of the hot embossing machine, it was rolled into a roll of 800 μm in diameter and placed into a hot embossed cavity

of polypropylene (PP Moplen HP500 N, LyondellBasell). On top of the PP foil with the PSU roll, a second sheet of PP was placed and subjected to hot embossing at a temperature of 170°C and an embossing force of 70 kN. Under this condition, the two PP foils melt together to one polymer part with a closed cavity filled by the microstructured PSU roll. At both ends of the cavity of the PP part, a hole was drilled down to the PSU foil. Afterwards, the microstructured PSU roll was dissolved chemically by

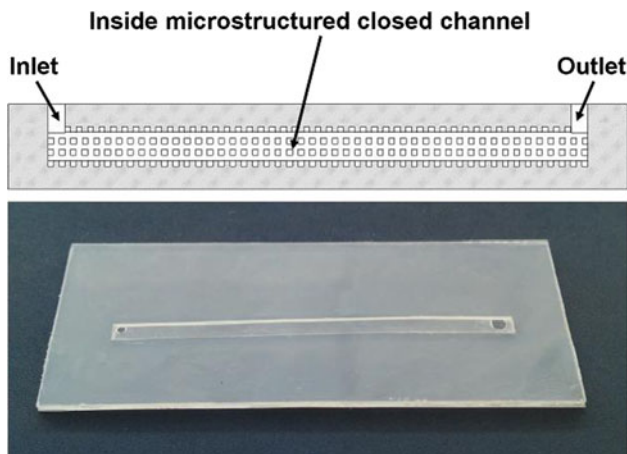


Fig. 16 Schematic sketch of a closed polymer channel with an inner structure (*top*) and replicated polypropylene part (*bottom*)

tetrahydrofuran (THF p.a., C_4H_8O , Marck KGaA) under ultrasonication for 1 h. The remaining PP part revealed a closed channel with a microstructured surface on each side and tight closure without any bonding difficulties. Without rolling the microstructured PSU foil, at least one side remained unstructured. Channels with an inner structure can be used afterwards for microfluidic or optical devices (Vannahme et al. 2010). Depending on the structure size and geometry, it is possible to manipulate the wetting behavior, cell adhesion, or interaction with blood or chemicals. In the optical device, the side wall structure influences light coupling and orientation into the channel and can be used for analysis. Due to the flexibility of hot embossing, also a metal wire can be used as support for foil rolling. The device may be applied as a flow sensor, because the flow in the channel changes the temperature and, hence, the electrical resistance of the wire. Figure 16 shows one of these PP devices with an inner structure, but without wire integration.

6 Conclusion

Different polymers were used to produce interstage mold inserts by hot embossing for subsequent replication of technical thermoplastics in the micro and sub-micro ranges. High-temperature polymers showed an excellent stability and replication accuracy even after 50 cycles of replication. Due to limited heating and cooling capabilities of commercial hot embossing machines, polymer combinations were analyzed for use in all common hot embossing machines. PC was generally found to be suited for replication to an interstage mold insert. However, success of the

complete process depended on the material combination of technical thermoplastic chosen. PSU showed good replication results and the stability test confirmed best values for temperature ranges below 220°C . At least twenty replications were performed without any significant loss of structure and geometry. The combination of polymers for the insert and embossed part is essential for good replication. Further development of polymers or compounds is aimed at enhancing the stability and increasing the accuracy even in the nanometer range.

Use of hot embossed polymer interstage mold inserts for microreplication significantly increases both the efficiency and throughput. New structures and geometries can be produced. Reduction of insert costs and improvement of productivity will provide for a wide applicability of optical devices or microfluidic systems.

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