### Electroforming of Tool Inserts for Injection Molding of Optical or Microfluidic Components

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#### Abstract

With a rapidly increasing international interest in "Lab-on-a-chip"-systems as well as affordable polymer optics, the combination of electroforming and injection molding offers an attractive fabrication solution.

Miniaturized analysis systems can be used for medical, security (anti terror monitoring) and environmental (waste water monitoring) applications. Optical components in polymer materials can be used for consumer electronics and for sensor systems.

The presentation will include the complete fabrication scheme for tool inserts based on machining and electroforming. Electroforming processes for nickel and copper will be disclosed. Processing parameters for the different types of tools as well as quality control measures will be presented.

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## Introduction

The heart of any injection molding process is the tool that determines the shape of the polymer product. Fabrication of the tool is traditionally done by one or a series of mechanical processes such as milling, electrodischarge machining (EDM), diamond turning or polishing. In case certain features of the tool - usually in the form of a tool insert, i.e. a smaller interchangeable metal part fixed in the larger molding tool - are very small (micro- or even nano-scale features) the traditional machining processes may be inadequate. In this case an effective solution can be utilization of electroforming using a suitable master as mandrel. The master can be structured in a number of different ways using photolithography, silicon micromachining, glass structuring or laser machining. After structuring of the master it must be made electrically conductive, either by electroless deposition of metal or by vacuum deposition processes.



*Fig. 1:* Injection molded disc in PMMA containing both refractive and diffractive optical components. The inlet is in the middle of the disc.

The use of galvanic processes to produce tools for injection molding of diffractive optical components, is a well established technology primarily driven by the optical media marked<sup>1</sup> for compact discs (CD) and digital video discs (DVD).

Similarly, although not quite as well known, electroforming and injection molding have been used to produce various refractive optical components such as contact lenses and low-cost lenses for automotive and consumer electronic applications (CD players, toys, etc.). Electroforming is also used to fabricate inserts for microinjection molding of high precision polymer components using the so-called LIGA-process<sup>2</sup>.

One of the main objectives of the work presented here is to demonstrate that with carefully selected processes it is possible to fabricate tools for injection molding of both diffractive and refractive optical components.

Another objective is to introduce a combination of laser machining of suitable polymer materials, combined with electroless metallisation and electroforming as an alternative route for fabrication of a durable tool insert for injection molding.

## **Experimental**

The processes used for fabrication of a particular tool insert will naturally depend on the application in mind. In the following two fabrication schemes are presented, one for simultaneous replication of both refractive and diffractive optical components, and one for replication of microfluidic devices (typically consisting of a micro-scale channel system with various inlets, mixer structures, etc.).



Fig. 2: Schematic cross-sectional view of the metal sandwich before the aluminum is dissolved.

Although the two fabrication schemes have been developed for a specific application, portions of each scheme may in some cases be successfully applied to other fabrication schemes as well.

## **Optical applications**

The fabrication procedure is based on an 8 mm thick aluminum disc onto which selected optical components (gratings and lenses) in glass are mounted. For each of the glass components a corresponding hole is machined into the aluminum disc. This ensures that only the optical surface is exposed and leaves room for the glue that holds the components in place. Using physical vapor deposition (PVD) a thin conducting layer of gold is deposited on the entire surface. On this substrate 50  $\mu$ m (1.97 mil) of stress-free nickel<sup>3</sup> or nickel-cobalt alloys<sup>4</sup> and

 $3000 \,\mu\text{m}$  (118 mil) of copper<sup>5</sup> is deposited using electroforming processes.

Various types of mechanical machining are then performed while the delicate optical surfaces are buried/sealed in the metal sandwich. The machining includes polishing of the back of the sandwich (with respect to the aluminum substrate), drilling of a center inlet and holes for the ejector pins.

The aluminum is then dissolved in warm sodium hydroxide (and the glass components are carefully removed). Finally, glue residue is dissolved and the thin gold layer selectively etched (Entreat 100, 50 g/l at room temperature).

The finished tool insert is then placed in a family mould that is mounted in the injection-molding machine (an Engel Victory HL650/130) and fixed using mechanical means.

The family mould has some characteristics that are not common for injection moulds:

- the entire mould is produced in beryllium-copper for maximum heat conduction in combination with high strength
- the mould is electrically heated and can be cooled conventionally with liquids or gas
- the entire cavity can be evacuated of air<sup>6</sup>

Injection molding using polymethylmethacrylate (PMMA, Plexiglas 7N OQ, Röhm), polycarbonate (PC, Makrolon test product 1265, Bayer AG) and cyclic olefinic copolymer (COC Topas 5013, Ticona) was performed at various process conditions, in order to establish which of the various parameters involved that influences the replication quality the most. AFM measurements of the replication quality of a diffractive grating were used for most of the evaluation, but other components on the test tools where examined as well.

#### **Microfluidic applications**

The usual approach for making a metal tool insert for microfluidic systems, is to apply electroforming of nickel to a substrate or mandrel consisting of a silicon wafer in which the desired channels has been etched<sup>7</sup>. We have mimicked this approach using laser machining of a polymer wafer instead of reactive ion etching of silicon (see Fig. 2).

There are advantages and disadvantages for both methods. The silicon approach clearly has the best resolution, but it is also the slowest and most expensive machining process. Laser machining offers the possibility of different channel depth on the same wafer, while this almost impossible

with micromachining of silicon. Both processes suffer from potential problems with the initial metal layer (either sputtered Ti/Au or electroless Cu), due to non-vertical sidewalls in case of silicon or carbon dust in case of ABS substrates.

Since laser machining requires no mask, and is a relatively fast and inexpensive method, it is in our opinion the obvious choice unless the microfluidic system in question has some extreme demands to the accuracy of the tool insert.



*Fig. 3:* Fabrication schemes for tool inserts that can be used for injection molding of microfluidic components. The silicon-based approach is adapted from the DEEMO method<sup>7</sup> while the ABS based method is covered by<sup>8-9</sup>.

The polymer wafer (ABS, NOVODUR P2H-AT natural, Bayer AG) is thermoformed into plates 6 mm in thickness and 4" in diameter. Acrylonitrile-butadien-styrene co-polymer (ABS) is chosen, since reliable electrochemical procedures for metallisation of ABS exist. A computer controlled excimer laser is used to machine the polymer.

After ablation the ABS plate is rinsed in ethanol and water. Then the butadiene phase is etched for 10 minutes at 63°C (145°F) in a mixture of chromic acid and sulfuric acid. After this, the plate is rinsed again and residual acid is reduced.

The etched structure is activated for 2 minutes in a mixture of Cataposit 958 (Shipley), NaCl and HCl followed by several rinsing steps.

Electroless copper is deposited in a Circuposit 3350 bath (Shipley) at 45°C (113°F). A continuos layer of 5  $\mu$ m (0.2 mil) copper is deposited in 60 minutes, and this thickness is sufficient for continued electroforming.

Copper is electroformed in an agitated and filtered copper bath. Pulse plating is utilized with 20 seconds cathodic deposition at 6 A/dm<sup>2</sup> (0.39 A/in.<sup>2</sup>) followed by 4 seconds of anodic stripping at the same current density<sup>5</sup>. The average current density is 4 A/dm<sup>2</sup> (0.26 A/in.<sup>2</sup>) resulting in a deposition rate of 0.8  $\mu$ m (31.5  $\mu$ in.) pr. minute. A total of 96 hours is used to reinforce the structure.

After electroforming has been completed, the ABS is dissolved in acetone and the copper disc is machined into a flat 4" tool insert.



*Fig. 4:* The surface (nickel side) of the tool insert with a diameter of 101.6 mm (4"). The insert holds two diffractive components (left side lenses) and two gratings (bottom right corner), the remaining components are plain glass pieces.

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# Results

The results of the project are an optimized electrochemical procedure for the fabrication of accurate electroformed tools, and a number of important findings regarding the conditions for the injection molding process required for exact replication.

## **Tools Fabrication**

#### **Optical applications**

After the first experiments with physical vapor deposition of gold onto the aluminum substrate (with embedded optical components), it became clear that the adhesion between aluminum and gold was insufficient. Consequently it was decided to deposit a copper layer of approximately 10  $\mu$ m (0.4 mil) from a pyrophosphate bath - after a zincate pre-treatment - on the aluminum substrate, followed by a 1 micrometer (40  $\mu$ in.) thick gold layer (Engold 2010C).



x: 20.0 µm

Fig. 5: AFM-image showing part of one of the gratings in the finished tool insert.

The glass components were glued (Loctite 358 UV-curable glue) into place on this smooth gold surface, and an adhesion layer of titanium (25 Å or 0.1  $\mu$ in.) and a conducting layer of gold (400 Å or 1.5  $\mu$ in.) was deposited by PVD.

In order to make it easier to machine the aluminum substrate (and because really pure aluminum is hard to get) a commercial aluminum alloy, containing silicon and copper as well as traces of many other elements, was used. This introduced some problems during the selective etching of the aluminum substrate in 1.5 mole/liter sodium hydroxide.

A cleaning step consisting of approximately 20 minutes in a 0.1 mole/liter EDTA solution at 95°C (203°F), effectively removed the black residues of the aluminum alloy, and exposed the copper layer.

The copper layer was selectively etched in an ammonium peroxide solution, with the machined copper back protected by tape.

At this point the glass components could be gently removed (if they had not fallen off by themselves) thereby exposing the "negative" gold/nickel surface of the tool insert. Finally both the electroplated and evaporated gold layers were dissolved in a commercial gold stripper (Entreat 100, 50 g/l).

#### **Microfluidic applications**

Although it is possible to ablate ABS using the UV excimer laser, some thermal effects are clearly seen because of a relative low absorption by ABS at the laser wavelength (248 nm). The thermal effects results, in extreme cases, in softening or collapse of the wall between adjacent micro channels - especially at high aspect ratios (when the depth of a channel is twice as deep or more compared to the width). Some soot or carbon dust will be left in the channels after laser machining, and in rare cases this carbon layer will prevent activation leading to problems for the electroless deposition of copper.

Future experiments will include laser machining of polyetherimide (PEI) and polycarbonate (PC) if suitable methods for metallisation and subsequent dissolution can be established.



Fig. 6: SEM micrographs showing some details of the pure copper tool insert. The tool had already been used for injection molding of polystyrene (PS) and polymer residues as well as discoloring (due to surface oxidation) is clearly seen.

Laser ablation of 150  $\mu$ m (6 mil) wide channels with approximately the same depth, proceeded without problems. After cleaning the wafer was metallised by electroless copper and a thick layer of copper was electroformed (either using Enthone CuproStar or pulse plating<sup>5</sup>) on top of the electroless copper layer. After machining and dissolution of the ABS substrate in acetone, the tool insert was ready for injection molding.

Since the ABS substrate is relatively soft, compared to metals or silicon, even small levels of internal stress in the deposited copper will lead to bending of the tool insert. Some investigations of stress levels in various copper electroforming processes in now being conducted, in order to minimize this problem. Severe bending of the tool insert will lead to a similar bending of the finished polymer components. For microfluidic systems this can be a serious problem, especially for gluing a lid on top of the channel system.

Introducing a layer of nickel (as for the optical tool inserts) before the copper layer, might reduce the problem since it is possible to deposit absolutely stress-free nickel and since a thick nickel layer would add considerable to the total stiffness of the substrate (ABS plus Ni).

## **Injection Molding**

#### **Optical applications**

The experiments have been performed as factorial experiments with the aim of determining which of the process parameters; melt temperature, mould temperature and injection speed, influence the replication quality the most. The results, expressed as the period and depth of the diffractive structure for PMMA, are shown in Tab. 1 (the results for PC and COC can be found in an article by Christensen<sup>10</sup>).

*Tab. 1:* Results of the factorial injection molding experiments for PMMA. The experiments were performed with vacuum in the cavity. For the most important parameter, the mould temperature,  $60 \,^{\circ}$  corresponds to the low value,  $100 \,^{\circ}$  is the medium value and  $121 \,^{\circ}$  is the high value.

Melt temp. – Mold temp. – Injection speed	Period ( $\lambda$ ),	depth,
	μm	nm
low - low - low	1,03±0,01	-
low – low – high	1,03±0,01	5-120
low – high – high	1,02±0,01	135
low – high - low	1,03±0,006	167
medium – medium- medium	$1,02\pm0,00$	64
high - low - low	$1,02\pm0,00$	40
high – low - high	$1,02\pm0,00$	5-20
high – high – low	1,02±0,00	152
high – high - high	1,02±0,00	123

"-" indicate that the value is not measurable

As it can be seen from the table, it is possible to perform precise replications of the period, i.e. replication in the plane is satisfactory in all cases. Replication in the 3<sup>rd</sup> dimension does however cause problems. Here, the process parameters are seen to influence the results. Mould temperatures have to be "high" (120°C in the case of PMMA) in order to achieve acceptable results in the depth. The temperature of 121°C is 10°C above the glass-transition temperature of PMMA and that is the most important result: the mould temperature has to be above the glass-transition temperature. Melt temperature and injection speed does not appear to influence the quality of the replications in the 3<sup>rd</sup> dimension.

Another important finding is that it is absolutely necessary to perform injection molding in a cavity evacuated of air. If not, the micron sized grooves that constitute the grating, will be filled with air and thus not molten polymer. Further more will the high pressure in some cases lead to combustion or burning of the polymer material (diesel effect).

Here, only results for PMMA have been presented. Similar experiments have been performed with PC and COC<sup>10</sup>. The results show the same influence of mould temperature and little or not influence of melt temperature and injection speed on replication in the depth. Especially for COC, but in some cases also for PC, replicated depths are far from satisfactory and it is concluded that of the three materials tested, PMMA is the most suitable for this purpose.

#### **Microfluidic applications**

In many ways the microfluidic systems are much easier to injection mold as compared to the optical components. At present small series of polymer components has been injection molded in polycarbonate (PC), polystyrene (PS), cyclic olefinic co-polymer (COC) and polymethylmethacrylate (PMMA).



*Fig. 7:* SEM micrographs of polystyrene components, injection molded using the copper tool illustrated in Fig. 6. The areas showed are approximately from the positions on the tool as the areas in Fig. 6.

# Conclusion

Electroforming using a combination of nickel (for wear and corrosion resistance) and copper (for thermal conductivity and easy polishing) is a powerful way of fabricating tool inserts for injection molding.

Using this process the original master structures (glass components or laser machined channels) were copied with an accuracy better than 10 nm (0.4  $\mu$ in.). After production of more than 1000 injected molded polymer components no wear or deterioration of properties or dimensions was observed.

It is absolutely necessary for the cavity of the injection molding tool to be evacuated, since any presence of air will lead to a reduction of replication accuracy and burned areas. The temperature of the mould surface must be higher than the glass-transition temperature  $(T_g)$  for a given material, in order to obtain the correct in-depth replication. A good replication in the plane however, is relatively easy to obtain even at lower temperatures

Utilization of laser machining, in combination with copper electroforming, was demonstrated as a relatively fast and inexpensive way to fabricate a tool insert for injection molding of polymer microfluidic devices.

Some problems associated with the choice of substrate material (ABS) and internal stress in the copper electroforming process (combined with the low mechanical strength of ABS) will be addressed in the future.



Fig. 8: AFM-image of an injection molded grating in PMMA (the image shows an area of 20 by 20 micrometers).

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