



# Fabrication and application of stainless steel stamps for the preparation of microfluidic devices



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## ABSTRACT

The use of an electrochemical polishing process to manufacture stainless steel stamps often gives rise to problems: there is a significant release of gas bubbles that can cause the photoresist mask to peel off. In this paper, a new procedure for the manufacture of stainless steel stamps is presented based on a combination of UV-lithography, gold electroplating, and electropolishing techniques. First, the pattern of the stamp is transferred to a photoresist layer by UV-lithography. The pattern in the photoresist is then filled in with a thin layer of gold formed by electroplating. The photoresist is then removed and the substrate etched in an electropolishing bath, with the gold layer serving as an etching mask. The steel stamps were repeatedly tested for the hot embossing of the polymethylmethacrylate substrate. The imprints were used for the preparation of functional microchips to test their functionality, the chips consisting of two polymethylmethacrylate plates: a plate with microchannels was embossed with a steel stamp, and a plate with inlet/outlet holes. The plates were joined by the thermal bonding method and the microchannels of the chip were then filled with a fluorescein solution to test whether the plates were properly bonded. The results indicate that this new procedure is suitable for the fabrication of flexible and durable masters for a roll-to-roll imprinting process.

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## 1. Introduction

Microfluidic devices are not only beneficial for analytical purposes, such as in chromatography [1], but also in many other fields as instruments for drug research [2], food analysis [3], molecular diagnostics [4] or cell culturing [5]. To enhance the wider use of microfluidic systems, it is important to reduce the production costs of microfluidic devices. Polymeric materials, such as polydimethylsiloxane (PDMS), polymethylmethacrylate (PMMA) and polycarbonate (PC), are usually used to simplify this production because they are easily processable and enable the mass production of microfluidic chips.

The processing of polymeric materials can be divided into two categories. The first category includes material removal by, for example, mechanical micromachining [6] and laser ablation [7]. Jáuregui et al. compared other material removal methods (micro-end milling, wire electro discharge machining/sandblasting and abrasive water jet) [8]. The preparation of a single chip is usually time consuming and, thus, the methods used are not entirely suitable for mass production. The second category makes use of

stamps to transfer the required microfluidic structures to a plastic substrate; for example, in the case of imprinting lithography [9,10], hot embossing [11,12] and injection molding [13]. Continuous UV roll imprint [14] and roll-to-roll nanoimprinting processes [15] belong to the group of techniques that utilize stamps for the mass production of microfluidic devices. Each imprinting technique requires a specific stamp type and/or material, but all stamps have to meet some general specifications.

Stamps should be durable as they have to withstand the effects of high pressure and temperatures during the imprinting (hot embossing) process without acquiring any mechanical defects. To ensure the easy removal of imprints from the stamps, the sidewall profile of the stamp structures should be conjugant (the top dimension should be narrower than the bottom dimension of the stamp structures). A smooth surface is important for any stamp as any roughness will be transferred to the plastic material during the imprinting process, which may lead to the defective bonding of the imprint to a plastic cover. Furthermore, surface roughness can cause difficulties during the demoulding process. Stamps made of a metal usually satisfy these conditions well.

Metallic stamps are commonly made by a combination of lithography and electroplating techniques [14,9]. Although UV-lithography restricts the dimensions of the finest structures to a

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micrometer scale, this process is much faster and the micrometer-scale is sufficient for many microfluidic systems. Nickel electroplated stamps are usually used for hot embossing [16], but also a stamp made of copper has been reported in the literature [17]. One disadvantage of electroplated stamps is that the electroplated metal not only fills the gaps in a photoresist, but also forms the entire body of the stamp and, thus, the structures must be over-plated. This over-plating process is time consuming (ca. 10 h) [17]. Without over-plating, the structures prepared by electrodeposition on a metal surface could tear from the metal surface during stamp removal. Over-plating increases the adhesion of the structures to the body of the stamp and thus it helps to avoid this problem. For some special applications, stamps made of epoxy resin [18], ceramics [19] or PDMS [20] were reported.

A new substrate removal approach was introduced by Chen et al. [21]. They prepared a steel stamp by a combination of UV-lithography and wet chemical etching. A stainless steel substrate was selectively covered with a photoresist while the uncovered area was chemically etched. The main advantage of etching is that this process is less time consuming because no over-plating is needed. The main disadvantage is that it makes the surface of both the stamp and its structures relatively rough. Cho et al. enhanced the etching process [22]. They prepared a steel stamp by a combination of UV-lithography and electropolishing, well-known surface finishing process [23]. However, during the electropolishing process there is a significant release of gas bubbles, which occurs on both electrodes. This may lead to the peeling of the photoresist from the substrate, and subsequently damage or destroy structures on the stamps' surface. We improved this process by replacing photoresist masking with gold layer masking. A gold layer is more durable and resistant to the polishing process. When a gold layer is prepared properly, it persists atop of the stamp's structures, which means that the stamp can be re-etched.

The main result of this contribution consists in enhancing the fabrication procedure of stainless steel stamps for the embossing of microfluidic structures. To determine the applicability of our proposed procedure, several experiments were performed, including the imitation of heat and tensile strain during a roll-to-roll imprinting process and applying the procedure to a mild steel substrate. The surface of the stamps was investigated using optical and scanning electron microscope (SEM) images. To confirm that the presented procedure is fully functional, the fabricated stamp was used for the preparation of a microfluidic chip.

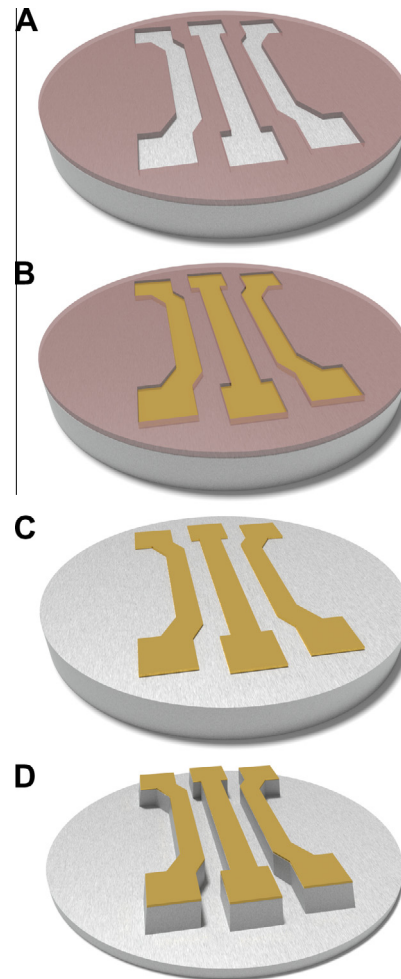
## 2. Experimental methods

### 2.1. Development and optimization of stainless steel fabrication

The main steps of stainless steel stamp production are illustrated in Fig. 1 and can be briefly described as follows: (A) A photoresist is spin-coated onto the surface of a cleaned steel foil (substrate). Then, standard UV photolithography is employed to create the pattern of the stamp microstructures in the photoresist. (B) A thin layer of gold is electrodeposited on the substrate. (C) The photoresist is removed and the gold pattern is used as an etching mask in the electrochemical polishing process. (D) The foil is etched in a polishing bath for a predetermined time until the stamp is ready to use.

#### 2.1.1. Stainless steel stamp fabrication

Circular pieces of stainless steel (Fe – 70.4%, Cr – 18.4%, Ni – 9.0%, Mn – 1.1%) foils with a thickness of 250  $\mu\text{m}$  and diameter of 6.35 cm, (PragoBoard CZ) were cleaned in an ultrasonic bath in a solution of Uniclean 155 10 wt.% (Atotech CZ) for 15 min at 50 °C and then electrochemically (both anodically and cathodi-



**Fig. 1.** Schema of the entire fabrication process of the stainless steel stamp: (A) preparation of stamp structures in a photoresist by standard UV lithography; (B) deposition of the gold layer on uncovered areas of the steel foil; (C) removal of the photoresist; (D) etching of the foil in a polishing bath.

cally) degreased in a solution of NaOH,  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_3\text{PO}_4$  – 40 g/l at 85 °C for 4 min at the current density of 6 A/dm<sup>2</sup>. After rinsing by DI water and drying, the foil was spin-coated with a ma-P 1275 photoresist (micro resist technology DE) until it formed a 7.5  $\mu\text{m}$  thick layer. Standard UV photolithography was used to create the pattern of the stamp microstructures. The photoresist was developed in a ma-D 311 developer (micro resist technology DE). Then a gold layer was deposited on the surface of the foil in areas uncovered by the photoresist. Gold plating on stainless steel usually requires special type of plating bath. An Auruna 311 plating bath (UMICORE Galvanotechnik DE) was specifically developed for direct gold plating onto the stainless steel substrate, but has low deposition rate (maximum of 0.2  $\mu\text{m}/\text{min}$ ) and the evolution of hydrogen can (in long-term deposition) damage the photoresist layer. Therefore the plating bath Auruna 311 was used only to deposit a seed layer at room temperature for 5 min at current density of 0.3 A/dm<sup>2</sup>. To make the fabrication procedure faster without damaging the photoresists layer, an Auruna 550 plating bath (UMICORE Galvanotechnik DE, 0.4 A/dm<sup>2</sup>, 3 min, at 50 °C) was used to finish the etching mask. After the plating, the photoresist was removed in the acetone bath and the surface of the foil was cleaned in 10 ml of concentrated  $\text{H}_2\text{SO}_4$  with 0.5 ml of  $\text{H}_2\text{O}_2$  for 30 s to remove the residue of any impurities. The back of the substrate foil (the side without gold plating) was covered by a polyester tape (type M42 by Europack Chrudim CZ). Then, the foil was etched in

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