Simple photolithographic rapid prototyping of microfluidic chips

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Abstract

We present a simple method for producing molds for casting PDMS microfluidic chips using photo-lithography with 35 mm photographic negatives as masks. We demonstrate the capabilities and limitations of the approach. We have optimized this procedure to create very good quality planar lab-on-chip structures and with this method can go from design to finished device in a day.

1. Introduction and motivation

Microfluidic/lab-on-chip technology has promising applications in many fields. There is broad interest in developing these systems amongst researchers from chemistry, biology, physics and engineering disciplines. Furthermore, as the technology becomes more mature there is an increasing desire to introduce the topic to undergraduate students [1]. In teaching or within research groups without access to sophisticated micro fabrication facilities the challenge is to manufacture microfluidic chips to a useful quality.

In microfluidic fabrication a common workflow is to pattern a photoresist such as SU8 on a silicon wafer or similar substrate and then perform a simple replica molding of this “master” by pouring PDMS (poly-dimethyl-siloxane) over the structure [2–5]. Alternatively the SU8 master may be used to produce a stamp suitable for hot-embossing into other polymer materials such PMMA or COC [6]. In either case, the SU8 is typically patterned using a photolithographic process.

The standard method involves preparing a chromium plated fused silica mask of the design and UV-exposing the SU8 coated substrate through the mask [7]. Properly optimized, the technique can produce high-resolution structures with smooth, vertical sidewalls. However, the preparation of the masks is a specialist task beyond the capabilities of many laboratories. Microfluidic devices rarely require sub-micron structures. Generally, channels of the order of tens or hundreds of μm are sufficient. Therefore, a lot of effort has been put into alternative methods for mask designs. These include ink-jet printing onto transparent polymers [8] or direct patterning of metal coatings [9]. Ink-jet printing is certainly the most straight-forward method of creating a mask directly from a CAD design, however, although it can nominally produce line widths down to 50 μm, edge quality is very poor (see again [8]) due to the nature of the printing mechanism and the contrast between light and dark areas is not sufficient for the longer exposures required for deep SU8 structures. Other methods include using photosensitive materials [10,11] or semi-transparent programmable screens [12,13].

In this paper we investigate the possibility of using conventional black and white photographic negative films as contact masks for photolithography. Analog film can have effective resolution equivalent to 5000 dpi [14]. However, the black areas on a photographic negative do not form a continuous film. In the clear areas, on the other hand, the polymer substrate of the film is more opaque in the UV than fused silica. Therefore, we can expect a poorer contrast in comparison to chromium masks. The granular nature of the film can also be expected to produce less abrupt edges between exposed and unexposed areas. In this paper compare several commercially available films and developers to overcome these limitations and produce useful microfluidic chips. We prepare SU8 test-structures and compare them qualitatively using SEM. We also examine the optical properties of the films to determine the important film properties for producing high quality molds. Finally we prepare microfluidic PDMS chips from the molds.

2. Materials and methods

2.1. Photographic negatives

Photographic film consists of a transparent polymer substrate coated on one side with gelatin. Silver halide crystals are
embedded into the gelatin. Exposure to light sensitizes the halides by releasing silver ions. Chemical development converts sensitized grains to metallic silver. Further processing, or “fixing”, removes the un-converted halides. Grains are generally of the order 200–600 nm in size and are randomly distributed through the gelatin layer, itself a few hundred micrometers thick. Film resolution is dependent on the size of the grains. Fine-grained films are referred to as “slow” as they require more light and so longer exposures to produce an acceptable density of silver particles [14].

We selected three commercially available films, Agfa Copex Rapid (AGFA), Bluefire Police (BF) and Rollei ATP. These are all marketed as high-resolution low speed films. Bluefire is also available under the trade names Adox CMS 20 and SPUR Ortho Pan UR.

Film contrast and resolution is strongly dependent on developer, therefore we used several different developers. Bluefire Micro Developer (MD) is designed for Bluefire police and is a hydroquinone based microfilm developer. Rollei DC is a “document reproduction” developer and SPUR Modular UR is a general high-resolution developer. Finally Ilford Microphen (MP) is also a popular high resolution developer.

For film investigation and comparison a test structure was defined using a commercial vector-graphics program (Dessault Systems, DraftSight 11.3). In the software the structures and line widths were drawn on a 1:1 scale. The image was then scaled and printed on A1 sized paper with a resolution of 1200 dpi using an HP Design Jet T1100 printer. The design includes a simple grid of dots of various sizes to 100 μm wide channels as well as a number of test structures including curved lines, crossing lines, and dots of various down to 10 μm. Large blocks of black and white were also included for transmission measurements.

The structure was designed to fill a full frame of the 35 mm film (36 × 24 mm). Alignment grids in the corners of the film were used to determine the correct positioning of the image within the camera viewfinder. A range of pictures were taken, aligning the viewfinder different grid lines and the resulting negatives measured. The alignment mark producing dimensions closest to the design size was used for the actual experiments.

The pictures were taken with a Pentax MZ30 SLR camera with 80 mm lens. The camera was mounted on a tripod and a delay shutter used to eliminate camera-shake. The design was photographed under controlled lighting at a range of exposures from 2 stops below to 3 stops above the level determined by the camera’s internal light meter for correct exposure.

The films were developed in our clean-room, according to the manufacturers specifications for the films and developers. The films are submerged in the developing agent for 5–12 min. The developer is then replaced by an acidic “stop bath” (Ilfordstop) which terminates the development process. Finally they are placed in a “fixing” agent (Ilford rapid fixer).

A combination of different films and developers are investigated, resulting in a total of six different films, each with 24–36 different negatives. Only combinations of film and developer for which established development conditions were available from manufacturers and other sources were tried.

2.2. SU8 master production

Transmission measurements on the negatives were made at a wavelength of 365 nm with an optical microscope (Nikon Eclipse ME600) and spectrometer (OceanOpticsMaya 2000 Pro.). For each film the intensities were normalized with respect to the lamp intensity at 365 nm. Transmission was measured in both clear and dark areas of the negatives and used to calculate the contrast.

The highest contrast negatives for each film/developer combination where used to fabricate SU8 structures. High viscosity SU8 (MicrochemSU8-2050) was spin-coated onto a silicon wafer. The negative was placed on top of the wafer and loaded into a Süss Microtech MA150 mask aligner, pre-loaded with a blank quartz plate in place of the normal chrome mask. When loaded the wafer is pressed against this plate, flattening the negative to the SU8 surface.

Standard SU8 exposure times were extended according to the measured clear transmission of the individual negatives to ensure full exposure. The SU8 films were developed and hard-baked at 150 °C for 10 min. The structures were investigated using SEM (Hitachi S-4800).

2.3. Preparation of PDMS microfluidic chips

To investigate the quality of the produced SU8 structures, the silicon wafers were cut into 26 × 38 mm pieces and used as molds in a PDMS replication procedure. The silicon masters were placed at the bottom of a stainless steel mold and liquid PDMS (Sylgard 184) was poured over them. The PDMS was cured at 100 °C for 1 h and released from the SU8 structures. The molded channel structure was sealed using a thin PDMS sheet activated by oxygen plasma.

3. Results

3.1. Film contrast

Transmission measurements of the mask design were obtained for the six different films and for all light exposures (Fig. 1).

All combinations show decreasing transmission with increasing light exposure since more light will lead to more silver crystals in the film. The AGFA + MD and AGFA + MP show a particularly strong dependency on exposure time. As well as exposure time, the transmission of the film is also dependent on transmission of the substrate polymer itself, and the development procedure.

While the UV-transmission of the polymer base is fixed, the development is clearly a very sensitive step. Comparing the three AGFA film combinations there is up to 40% difference which is attributed to the development procedure. Clearly the SPUR and MP developers have “overdeveloped” the film. This also accounts for the difference between the two Rollei films. The Bluefire Micro-Developer (MD) shows the best overall results regarding UV-transmission for both the AGFA and BF films.

A large variation is also observed in the dark areas (Fig. 1, top). Transmission ranges from 72% for BF to below 0.1% for AGFA, Bluefire and Rollei. Furthermore, some films are less sensitive and maintain similar values for all light exposures, while others (like the BF film) span a wide range.

The ratio of transmission in clear to dark areas was taken giving the contrast Fig. 2. We see a large contrast difference, across the different film/developer combinations and exposure times. Generally, a longer light exposure results in better contrast. Very high contrasts (over 1:300) are recorded for both BF and AGFA films (developed in both MD and MP). The Rollei ATP and MP combination also results in nice contrasts (almost 1:1000) whereas, contrast below 1:10 is observed for both the Rollei + DC and AGFA + SPUR combinations.

3.2. Photoresist quality

All SU8 structures were developed on SU8 layers with a nominal thickness of 100 μm. Bluefire Police film and Agfa Copex Rapid both developed in the Bluefire Micro developer produced the best defined structures. In both cases the 100 μm wide microfluidic test channel was clearly reproduced and very stable following hard-
baking. However, looking at the test structures (Fig. 3) we can clearly see some of the limitations of this approach. Firstly the walls of the SU8 show a clear “wrinkled” effect. Furthermore, all structures appear to be significantly undercut.

For free-standing structures such as those used to mold channels, this resulted in the loss of structures smaller than approximately 40 or 50 \( \mu \text{m} \) depending on how well the feature is supported by surrounding material (Fig. 4, top). For inverse structures however the undercutting leaves well defined holes and trenches in the SU8 (Fig. 4, bottom). It would appear that the finer features are properly cross-linked at the UV exposure stage but that they are lost during development.

All structures show a clear tendency to produce overhangs at edges (Fig. 3) and surface debris is often left on the silicon substrate after development. As the black masking areas of the negative are not are continuous film and not completely opaque to the UV, cross linking begins in all areas of the film as soon as exposure begins. SU8 cross-linking commences at the surface and works down toward the substrate. Thus, exposure must continue until the areas under the “clear” parts of the negative have been completely exposed. If contrast is poor, the areas under the “black” parts of the negative may be significantly exposed also. The partially exposed surface will cure and produce debris during development. Furthermore, since the edges of features on the film are not

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**Fig. 1.** UV transmission of clear (bottom) and dark (top) areas of negatives as a function of exposure.

**Fig. 2.** UV transmission contrast ratio of clear to dark areas of negatives.

**Fig. 3.** SEM of SU-8 line structures formed using Bluefire Police negatives developed in Bluefire Micro developer and 2 stops over exposed. The wafer was cut after SU-8 development to show cross-section.

**Fig. 4.** SEM of “grid” test structure from same sample as Fig. 3 (top: raised, bottom: indented).
perfectly abrupt we can also expect a graduation of UV exposure from black to white. This produces both the undercut walls and the overhanging surfaces seen. It may also be responsible for the wrinkled texture of the walls.

However contrast is not the whole story. Consider the fully exposed Agfa film developed in Microphen and compare with Bluefire Police or Agfa Copex two stops over-exposed and developed in the Bluefire Micro developer. All have the same contrast of approximately 300:1. However the Agfa/Microphen combination produces an unusable SU8 structure in comparison with the other two films. The principal difference is that the Agfa/Microphen has considerably lower transparency in the clear areas compared with the other negatives. This requires considerable increase in exposure time. As well as prolonging exposure in the dark areas this may also lead to surface heating of the SU8 which is also known to induce partial curing [15].

3.3. PDMS structures molded from SU8 masters

Several replications of the SU8 structures have been successfully realized in PDMS (from both BF and AGFA negatives). The PDMS replicates all features in the SU8, including overhangs, holes and surface structures down to below 1 μm. Furthermore the soft PDMS releases without any problem from the SU8 structures and requires no use of additional release agents or surface coatings. The hard-baked SU8 masters are also very durable and some structures have been replicated up to 30 times in our lab without any visible signs of wear or feature reduction.

4. Conclusions

Bluefire Police and Agfa Copex Rapid films were able to produce SU8 masters of sufficient quality for microfluidic device production. Both films were characterized by high contrast and exceptionally clear underlying base. The results required a developer designed specifically for high contrast work such as those used for document reproduction like Bluefire Micro developer. SU8 masters were durable and multiple PDMS chips could be produced from them. In all, once a workflow has been established, it is possible to go from initial design to SU8 master of multiple designs in a day’s work. The method is especially useful for rapidly trying many designs at a low cost. It also lends itself to teaching applications where students can design and produce their own microfluidic chips with reasonable time to good quality. However, the method is limited to larger features (greater than 50 μm) and low aspect ratios (1:3) and great care must be taken to optimize the film development process to produce the best results.

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Appendix A. Supplementary data

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References