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Multi-layer SU-8 lift-off technology for microfluidic devices

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Abstract

This paper reports on a novel multilayer SU-8 lift-off technology which allows for low cost rapid prototyping of microfluidic devices. The process presented is based on a multi-layer structure of SU-8 which can be released from the substrate after processing and enables the creation of through holes. The lift-off is accomplished during the development by making use of the volume shrinkage of the SU-8 during postbaking and by modification of the adhesion to the substrate. To demonstrate the technology, prototypes of a multichannel microdispenser according to the Dispensing Well Plate (DWP^{TM}) principle (Koltay *et al* 2004 *Sensors Actuators* A **116** 472, 483) were fabricated. The samples contain 24 parallel dispensing units with 100 μm through holes and a dosage volume of 60 nl. For the first time all functional structures such as reservoirs, channels and through holes (nozzles) of the DWP^{TM} were realized exclusively in the photodefinable epoxy SU-8. To assess the quality of the SU-8 process the geometry of the presented prototypes is characterized by profiler measurements and scanning electron microscopy. Furthermore, the dispensing performance is studied experimentally by gravimetrical measurements. A reproducibility of the dosage volume of 1% and a homogeneity within individual droplet arrays of 3.6% were achieved.

1. Introduction

Early microfluidic systems as reported in [3] have typically been fabricated from silicon or glass by micromachining and bonding technologies. Although these technologies provide high precision and good yield, the costs for material and production render them uneconomical if large area devices have to be fabricated. Furthermore, in many medical and pharmaceutical applications disposable devices are mandatory which particularly requires the use of low-cost materials and processes. Disposable devices are cheap to produce in large volumes by polymer replication technologies such as injection moulding or hot-embossing. These technologies are well suited for large volume production; however, for design, test, prototyping and production of small quantities they are too expensive due to their high setup costs. Furthermore creating small through holes in a highly parallel way as required in some applications is still a challenging issue with polymer replication techniques.

Alternative fabrication methods based on SU-8 have been investigated in the past [4]. SU-8 technology is compatible with standard silicon processing conditions. Also multilayer structures can be patterned with standard mask aligners. The technology provides the feasibility of creating high-aspect-ratio features greater than 1:18 [5] and vertical sidewalls of up to 2000 μm [6]. The applicability of SU-8 as a functional material for MEMS was demonstrated before [4, 6]. In particular SU-8 can be applied as a material for microfluidic structures being stable against dimethyl sulfoxide (DMSO) and many other solvents [7–10]. Therefore SU-8 is an ideal material for rapid prototyping in microfluidics. This is in particular demonstrated for the Dispensing Well Plate (DWP^{TM}) used as a working example for the presented SU-8 lift-off technology in the following.

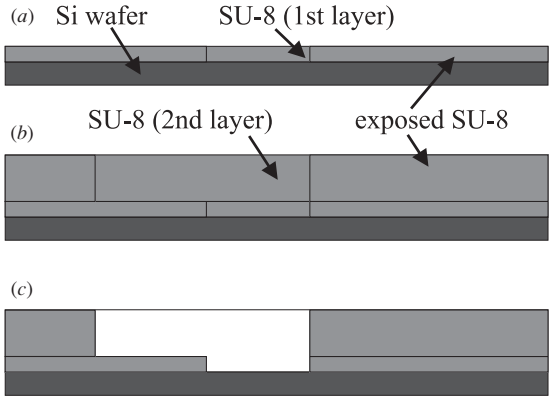

Figure 1. Principle of the multilayer SU-8 process: (*a*) first layer prebaked, patterned and postbaked, (*b*) second layer prebaked, patterned and postbaked, (*c*) simultaneous development (see text for details).

2. Technology

2.1. Multilayer process description

The SU-8 photoresist allows for the realization of multilayer structures by the successive processing of several SU-8 layers—deposition, prebaking, exposure and postbaking—on top of each other and a following simultaneous development. Due to the fact that SU-8 is a negative photoresist this technique has the limitation that no sealed structures can be realized directly. Several approaches to overcome this limitation are described in the literature, i.e. the use of sacrificial layers [5] or the sealing of SU-8 structures with glass [11]. In the approach presented here, we focus on the fabrication of through holes in the SU-8 and a lift-off process to release the fabricated structures but not on sealing.

The principle of this multilayer process is sketched in figure 1. In our experiments we use Nano^{TM} SU-8 100 (Microchem Corp., MA, USA) and 100 mm silicon test wafers. The process steps were carried out using a RC5 Gyrset spin coater, a MA6/BA6 mask aligner (both Karl Suess Wetzlar GmbH, Germany) and conventional hotplates. The multilayer process starts with the deposition of the first SU-8 layer on a silicon substrate by spin coating. With SU-8 100, layer thicknesses from 30 μm to 600 μm are achievable on silicon. For layers with thicknesses up to 250 μm the resist is spread at 300 rpm with an acceleration of 100 rpm s^{-1} for 10 s. The spread cycle is followed by the spin cycle of 30 s. The spin speed depends on the desired layer thickness; the acceleration is 300 rpm s^{-1}. When spin-coating layers that are thicker than 250 μm, the thickness depends not only on the spinning speed. Due to the high viscosity of the SU-8 100 and the low rotational speed the resist just spreads on the substrate. Thus not only the spinning speed but also parameters such as the dispensed volume and the dispensing position become important. Therefore the spin cycle time was extended to 60 s to minimize the influence of these factors. Furthermore the substrate is rotated at 20 rpm while depositing the resist to create a centred start volume. This procedure leads to better reproducibility of the layer thickness from wafer to wafer.

After spin coating, an extended prebake is performed at 95 °C. The heating decreases the viscosity by increasing the mobility of the polymer molecules. Thus gravity can lead to the annihilation of the edge bead and other possible variations of the layer thickness. To obtain a homogeneous layer thickness, it is important to prebake the SU-8 on a plane and accurately adjusted horizontal hotplate. During the prebake the solvent contained in the SU-8 evaporates, which causes an increase of the viscosity of the SU-8 and a hardening of the layer. Subsequently the temperature is ramped down under the glass transition temperature. Especially between 60 °C and 50 °C the ramping allows the polymer molecules to re-crystallize in a stress-free way. The duration of the prebake strongly depends on the layer thickness. Therefore layers of several hundred micrometres require up to several hours before the solvent is evaporated. The prebake temperature can be increased up to 130 °C to speed up the baking time.

After the prebake the first layer is patterned by a standard UV-lithographic process with an exposure dose as specified in the data sheet of the resist. To prevent the resist from overheating, possibly causing mask sticking problems, the exposure dose is applied in steps of 50 mJ cm^{-2}. A waiting time of 20 s allows the SU-8 to cool down. In contrast to single layer SU-8 processes only a short post-exposure bake (PEB) is performed afterwards at 65 °C. The reason for this is that the prebake of the next layer induces further polymerization in the exposed photoresist areas below. The short bake prevents diffusion of the solvent from the subsequently spin-coated layer to the previously created one (figure 1(*a*)). Afterwards the next layer is spin coated (figure 1(*b*)). Note that the layer thickness not only depends on the spinning speed but also on the substrate surface; i.e., spinning SU-8 on top of SU-8 requires another speed than spinning on silicon. The speed required for SU-8 surfaces is approximately 1.2 times higher than that for silicon surfaces. Depending on the thickness of the first layer, it might be necessary to perform the prebake of the subsequent layers on a hot plate with a cap or in a convection oven. Otherwise the prebake time for thick sub layers must be increased due to the low thermal conductivity of SU-8.

For prebaking single SU-layers the ramping of the temperature is not critical, because the free migration of the polymer molecules beyond the glass transition temperature avoids stress being induced in the layer. However, temperature ramping becomes important in multilayer processes. The layers underneath the actually processed one have been exposed before, and therefore these layers continue to polymerize and do not reflow. The mismatch between the thermal expansion coefficients C_{th} of the cross-linked SU-8 (52 ppm K^{-1}) and the substrate (e.g. silicon, $C_{\mathrm{th}} = 3$ ppm K^{-1}) can therefore cause adhesion problems. Thus the temperature is ramped up from room temperature to 95 °C at 10 °C min^{-1} and cooled down at 2 °C min^{-1}. After the exposure a PEB is performed with the same temperature ramps as the prebakes. Afterwards more layers can be deposited. In principle any number of SU-8 layers can be processed on top of each other. Finally, all layers are developed simultaneously in MR-Dev-600 (Micro Resist Technologies GmbH, Germany) and rinsed in 2-propanol (figure 1(*c*)).

2.2. Lift-off process description

In order to release large SU-8 structures, a novel lift-off technology based on OmniCoat^{TM} (Microchem Corp., MA,

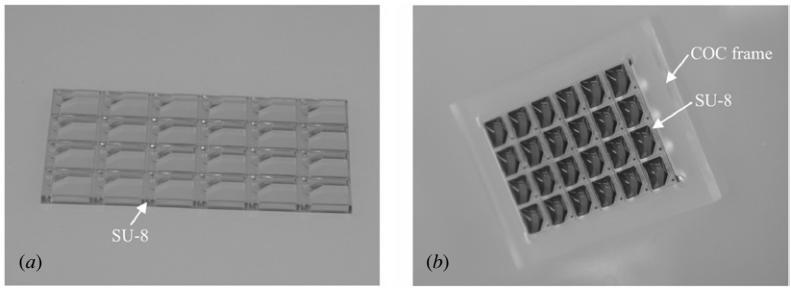

Figure 2. Photograph of a Dispensing Well Plate (DWP™) (*a*) after lift-off, (*b*) mounted into a COC frame and filled with coloured DMSO.

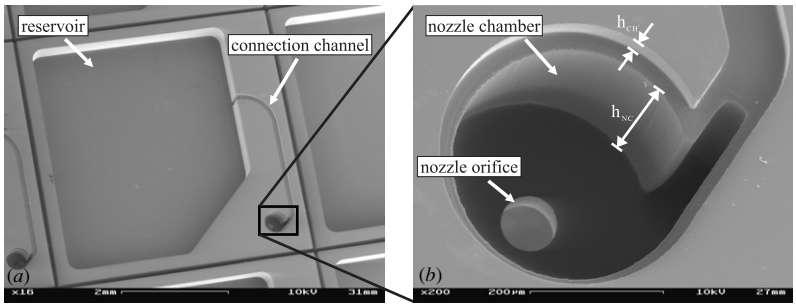

Figure 3. SEM of (*a*) a single dispensing unit of the DWP™ fabricated by SU-8 lift-off technology, (*b*) close-up of the nozzle.

USA) has been established. OmniCoat™ has been used as a removal layer for SU-8 in UV-LIGA before, because OmniCoat™ can be developed selectively against SU-8. However, it is generally not possible to remove large functional structures by etching the OmniCoat™ [12]. OmniCoat™ layer thicknesses of less than 100 nm provide only a very small gap for the developer and do not allow for dissolving the layer below extended structures. To overcome this problem a different property of OmniCoat™ is utilized: though being an adhesion promoter for metals and quartz, OmniCoat™ in fact *decreases* the adhesion of SU-8 on silicon. We observed that by varying the spinning speed and therewith the OmniCoat™ layer thickness the SU-8 adhesion on silicon can be varied over a certain range. The thicker the OmniCoat™ layer the lower the adhesion between SU-8 and silicon. If the adhesion is weak enough, stress in the SU-8 can cause the SU-8 layer to peel off. The cross-linking process within the SU-8 during curing causes such a stress at the silicon-SU-8 interface due to the volume shrinkage of about 7.5% of the SU-8. The stress induced at the material interface increases with the lateral dimensions and the height of the SU-8 structures. Due to the lowered adhesion the SU-8 structures are released from the substrate during development if the OmniCoat™ layer is suitably chosen. The whole release process can be accelerated by using an ultrasonic bath.

The described process works without removing the OmniCoat™ between the silicon and the SU-8 structures and therefore is not considered to be a sacrificial process like that studied in [12]. Due to the fact that the magnitude of the stress induced at the material interface depends on the structure shape, size and height, no general advice can be given as to which OmniCoat™ thickness indeed leads to a release of a given structure. The adhesion rather has to be tuned by varying the OmniCoat™ thickness to accomplish the release of the structure at the end of the process. However, the use of silicon as a substrate is essential; the process does not work on metal or glass substrates. These surfaces provide a lowered adhesion anyway and the SU-8 is likely to peel off unintentionally during processing.

The OmniCoat™ processing on a 100 mm wafer is as follows. First 2 ml of OmniCoat™ are spin coated onto the substrate (spread cycle: speed: 500 rpm, acceleration: 100 rpm s^{-1}, time: 5 s; spin cycle: speed: 1000 to 5000 rpm, acceleration: 300 rpm s^{-1}, time: 30 s). Then a baking step is performed at 200 °C for 1 min to vaporize the solvent. In comparison to other lift-off technologies, this technique has several advantages. The use of photoresist as a sacrificial layer is time consuming due to spinning, baking, rehydration and exposure steps. Using metals as a sacrificial layer, the deposition becomes expensive. In both cases the sacrificial layer has to be dissolved through a small gap which can be—depending on the material—very time consuming or even impossible below large structures [12]. In contrast OmniCoat™ is deposited within 2 min and does not need to be removed through small gaps by extended developing.

3. Application

3.1. The Dispensing Well Plate (DWP™)

The Dispensing Well Plate (DWP™) method has been developed for the highly parallel, simultaneous and contact-free dispensing of reagents into micro well plates used in pharmaceutical research [1, 2]. A DWP™ microdispenser with 24 dispensing units made of SU-8 is displayed in figure 2. Figure 3 shows a scanning electron microscopy (SEM) of a single dispensing unit. The working principle described in detail in [1] is then as follows. The reservoirs are loaded with different reagents. Capillary forces transport the reagents from the reservoirs to the micro nozzles without any need for

external actuation. The nozzles have a defined geometrical volume. By applying a pneumatic pressure pulse to the whole upper surface of the DWP^{TM}, the liquids contained in the nozzles are ejected as free-flying jets. The dispensed volume is essentially defined by the geometrical volume of the nozzles and not by the actuation. It is almost independent of the amplitude and the duration of the actuating pressure pulse. The dispensing process and the influence of the system parameters on the performance are studied in detail in [2].

3.2. Prototype description

Using the SU-8 lift-off process described before, DWP^{TM} microdispensers as displayed in figure 2 were fabricated. The prototypes have lateral dimensions of 27 mm × 18 mm and a height of about 551 μm. They contain 24 dispensing units. Every unit consists of a reservoir which is connected via a capillary channel with a through hole, the nozzle. The nozzle consists of a nozzle chamber and a nozzle orifice (see figure 3). The nozzle orifices have diameters of 100 μm and are realized in a 63 μm thick SU-8 layer. Reservoirs and nozzle chambers have a height of 416 μm. The connection channels have a cross-sectional area of 72 μm × 100 μm.

A common problem when processing ultra-thick SU-8 microstructures like the 416 μm high reservoir layer is the large internal stress of polymerized SU-8. Even if adhesion problems do not affect the multilayer lift-off technology, the internal stress can still lead to cracking of the patterned structures. Even bending of the silicon substrate has been observed while processing layer thicknesses of several hundred micrometres. Whether cracking indeed turns out to be a problem depends essentially on the detail of the geometric structure. To prevent cracking due to internal stress in the thick reservoir layer the DWP^{TM} is separated into discrete dispensing units by trenches acting as stress-relief structures. Only the 63 μm thin nozzle layer builds a cohesive film. The other layers are split into 4.5 mm × 4.5 mm sized elements by lithography. Thus the stress required for the final lift-off is mainly induced by the 63 μm thin nozzle layer.

3.3. Prototype fabrication

The process sketched in figure 4 starts with the deposition of an OmniCoat^{TM} layer to reduce the adhesion as described before (*a*). 2 ml of OmniCoat^{TM} are spin coated onto a 100 mm silicon substrate (spread cycle: speed: 500 rpm, acceleration: 100 rpm s^{-1}, time: 5 s; spin cycle: speed: 4000 rpm, acceleration: 300 rpm s^{-1}, time: 30 s) and baked for 1 min at 200 °C. Then the first 63 μm thin SU-8 layer is deposited (spread cycle: 500 rpm, 100 rpm s^{-1}, 10 s, spin cycle: 3500 rpm, 300 rpm s^{-1}, 30 s) and prebaked at 95 °C for 30 min (ramp up: 10 °C min^{-1}, ramp down: 2 °C min^{-1}). This layer is exposed with a mask containing the nozzle orifices (exposure dose: 6 × 45 mJ cm^{-2}). A PEB of 10 min is performed at 95 °C (*b*). The second layer (416 μm) is spin coated at 600 rpm for 60 s (acceleration: 300 rpm s^{-1}) and prebaked at 95 °C for 5 h 30 min. The reservoirs and nozzle chambers are exposed with a dose of 16 × 46 mJ cm^{-2}. Postbaking for 45 min at 65 °C induces further polymerization (*c*). Afterwards the 72 μm thick channel layer is spin coated (spread cycle: 500 rpm, 100 rpm s^{-1}, 10 s, spin cycle:

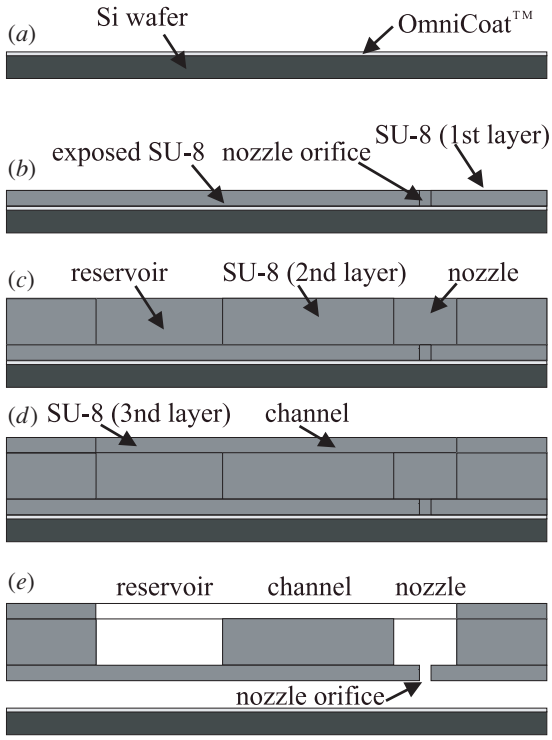

Figure 4. Description of the SU-8 lift-off process for the fabrication of the DWP^{TM} (see text for details).

5000 rpm, 300 rpm s^{-1}, 30 s) and prebaked at 95 °C for 30 min. The following exposure (dose: 5 × 46 mJ cm^{-2}) defines the channels and the upper parts of reservoirs and nozzles in the SU-8 (*d*). After a final PEB at 95 °C for 30 min, the three SU-8 layers are developed simultaneously in MR-Dev-600 (Micro Resist Technology GmbH, Berlin, Germany) for 2 h. Due to the lowered adhesion, and supported by ultrasonic power, the DWP^{TM} dispensing chips are released from the silicon substrate. After the development the chips are rinsed in 2-propanol for 10 min. The OmniCoat^{TM} remaining on the SU-8 surface is removed by MF-319 (Rohm and Haas Electronic Materials, Lyon, France). Finally, the SU-8 structures are rinsed in deionized water for 10 min (*e*).

It has been observed that the time from the process start to the final development in MR-Dev-600 greatly influences the quality of the development. If the processing steps described above are performed with a delay of several days between the individual steps, the nozzle layer might not be developed completely. Even if the development time is extended to 24 h SU-8 can remain in the nozzle orifices. Using exactly the same process parameters but carrying out the whole process without any delay (typically within 3 days) the whole DWP^{TM} structure is well defined and fully developed. As can be seen in figure 5, the created orifice is completely developed and exhibits a well-defined sharp edge after being released from the substrate.

After being released from the substrate the SU-8 DWP^{TM} chips are glued into a frame for stability reasons (figure 2(*b*)). The frame is made of Cyclic Olefin Copolymer (COC) by conventional milling and contains 1 mm through holes beneath the nozzles. The bonding is performed using a UV sensitive glue (Loctite 302) which is spray coated onto the COC. This

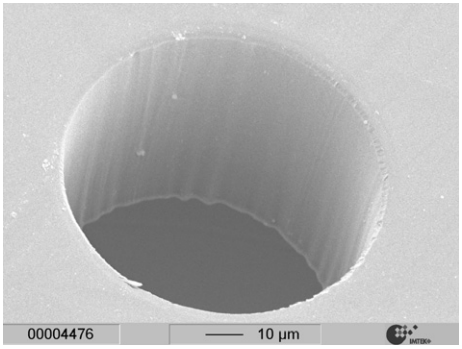

Figure 5. SEM of a nozzle orifice.

enforcement of the chips is necessary because during the operation of the DWP™ the whole upper surface is loaded with a pressure pulse of up to 0.5 bar. If not supported, the applied load would break the thin nozzle layer of 63 μm thickness holding together the individual dispensing units.

With a contact angle of about 75° between SU-8 and water, SU-8 shows an almost hydrophobic behaviour. To achieve a proper self-priming of the fluidic structures using aqueous solutions the SU-8 surface has to be modified. Therefore the fluidic structures were selectively coated with poly(*N*, *N*-dimethylacrylamid) to obtain a long-time stable hydrophilic surface.

4. Prototype characterization

4.1. Geometry

The dosage volume of dispensing systems according to the DWP™ principle is defined by the geometrical volume of the nozzle. Therefore the height of the SU-8 layers is especially critical. The distribution of the layer thicknesses over a DWP™ dosage chip has been measured to estimate the influence of the SU-8 multilayer technology on the DWP™ performance. The measurements have been carried out using a Tencor P-11 surface profiler. In each dispensing element the height of the third and second SU-8 layer h_{CH} and h_{NC} respectively (cf figure 3) was quantified. The measurement of the channel heights exhibits a mean value of 62.9 μm and a standard deviation of 3.0 μm. The mean height of the second layer (reservoirs and the lower part of the nozzle chambers) is 415.8 μm with a standard deviation of 9.0 μm. So the overall coefficient of variation (CV) of the nozzle chamber height h—and therewith of the nozzle chamber volume determining the dosage volume—is 2.0% across the dispensing chip. This corresponds very well to the measured homogeneity within a dispensed array as discussed in the next section.

4.2. Dispensing performance

To drive the DWP™ the prototype actuation unit described in [1] has been used. The dispensing performance of the presented prototypes was characterized by gravimetric measurements. This has been done by dispensing all 24 droplets on a microbalance. From the overall dispensed mass the mean dosage volume of each dispensing unit has been calculated taking systematic errors due to evaporation and adsorption into account. Figure 6 shows the reproducibility of the mean dosage volume per dispensing unit by a series of consecutive shots. The performance of the presented SU-8 prototypes in terms of accuracy is similar to the behaviour of previous DWP™ prototypes from silicon [13]. The measurements exhibit a mean value of 58.2 nl and a very good reproducibility of 1% within 70 shots, as shown in figure 6. Afterwards a trend of decreasing volumes can be observed. The reduction of the dispensed volume is attributed to the depletion of the reservoirs. The meniscus formed in the reservoirs as a result of the depletion generates a surface force which counteracts the proper filling of the nozzles. This effect has also been observed at former prototypes and is discussed in [14].

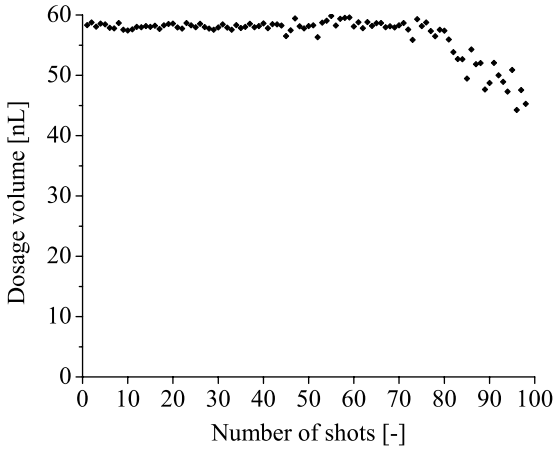

Figure 6. Reproducibility of the mean dosage volume per dispensing unit by a series of consecutive shots.

In order to derive the statistics of the distribution of the dispensed volume within one dispensed droplet array, measurements of every single dispensing unit have been conducted. Therefore, each dispensing unit has been filled consecutively with DMSO while all the others have been left empty. With only one nozzle filled 39 dispensing cycles have been performed and the measurement was repeated for each of the 24 nozzles. The whole data acquired by this experiment is displayed in figure 7. A statistic evaluation of the data reveals that the mean homogeneity of the dispensed droplet array of 3.6% is satisfactory. This figure was determined by calculating the CV for the 24 nozzles of each corresponding dispensing event (i.e. the normalized standard deviation for each row) and averaging the obtained CV values over the 39 individual experiments.

The described experimental conditions represent a worst case for the dispensing situation. Because only one nozzle is filled while all others are empty, the pressure pulse created on the upper surface of the DWP™ is suspected to be less stable and homogeneous. Thus it can be expected that by a proper filling of the DWP™ and a simultaneous dosage of all nozzles the homogeneity is at least as good as that determined in this experiment. Furthermore the experiment proves that the system is very robust against depletion of individual reservoirs and might also be used when not all reservoirs are completely filled.

Considering each nozzle for itself (i.e. statistic evaluation of each column) it turns out that the reproducibility of the

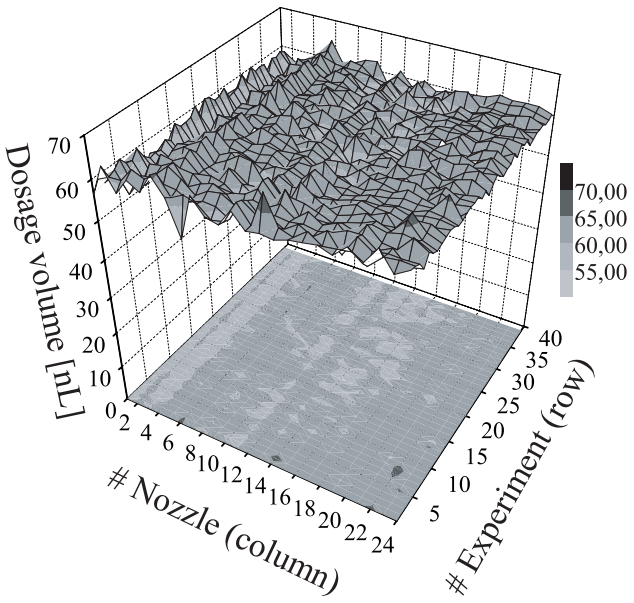

Figure 7. The dispensed volume of every nozzle of a DWP™ dosage chip for a series of 39 dispensing events (see text for details).

individual nozzles is good. The mean reproducibility of all nozzles determined by calculating the CV for every nozzle (i.e. for every column) and averaging over these 24 values results in a mean CV of 2.2%. The mean dosage volume of every nozzle can be obtained in a similar way by averaging over all experiments (i.e. column-wise) and calculating the mean value and CV of these average dosage volumes over all 24 nozzles. This results in a mean dosage volume of 60.9 nl with a mean CV of 2.2%. These figures prove that each nozzle for itself works in a very accurate and reproducible way, and that the good reproducibility as well as the mean dosage volume, which was determined in the first experiment by measuring the whole droplet array (all 24 nozzles simultaneously), is not due to statistical smoothing but stems from the equal and good performance of all individual nozzles. Especially the reproducibility of the mean dosage volume of 1% measured in the first experiment can also be deduced on the basis of the data displayed in figure 7 by calculating the CV over the averaged dosage volumes (i.e. calculating the mean value row-wise). This procedure results in a CV of 0.8% which is consistent with the first experiment.

5. Summary

A novel technology to lift off multilayer SU-8 structures from silicon substrates has been presented. This fabrication method especially allows for the creation of through holes, nozzles and orifices. The processing of the SU-8 multilayer structures and the lift-off technology based on OmniCoat™ was described in detail. The applicability of this technique for the fabrication of microfluidic devices has been demonstrated by producing highly parallel dosage chips according to the DWP™ principle. The DWP™ dosage chips were made up of three layers of SU-8 containing reservoirs, channels and nozzles and were released form the substrate after processing by the presented lift-off technology. The release of the structures from the substrate produced sharp and defined nozzles applicable for microdispensing. The characterization of critical dimensions exhibited a CV of 2.0% across the whole device.

The dispensing performance of the fabricated DWP™ dosage chips was finally characterized by gravimetrical measurements. The investigated DWP™ prototypes were able to deliver a fixed volume of about 61 nl with a CV as low as 3.6% within an array of 24 droplets and a reproducibility of about 2.2%. This proves that the presented SU-8 technology is comparable to silicon technology in terms of dimensional accuracy. Therefore the presented process is also applicable to systems where the dimensional stability and tolerances are critical.

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