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IMPROVING THE MANUFACTURING PROCESS OF MULTI-LEVEL MICROFLUIDIC DEVICES BASED ON THE LAMINATION OF SUCCESSIVE DRY FILM PHOTORESIST LAYERS

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ABSTRACT

The development of gas microfluidic devices have been generally restricted by the limitations of the microfabrication techniques. For years, the use of soft-lithography has been widely extended allowing the rapid prototyping of microfluidic devices in a multidisciplinary environment [1]. However, this technique is not adequate for complex structures, mass production or efficient integration. More recently, the development of the promising additive manufacturing techniques has allowed for the realization of 3D microfluidicdevices and a higher flexibility in the prototyping [2], with the most advanced laser stereolitography techniques achieving resolutions down to the sub-micron scale [3]. However, the cost of this technology is still elevated and it cannot be efficiently integrated for mass production. Therefore, the manufacturing of complex microfluidic devices with high resolution and low cost with fast processing keeps being a challenge with microfluidics remaining predominantly associated to a 2D technology.

Recent approaches for completing more complex microfluidic devices have involved the use of different photoresist materials combined with conventional photolithography techniques. SU-8 is a well-known liquid negative photoresist epoxy that is typically spin-coated over a substrate, but it is costly and requires significant know-how to achieve complex structures [4]. Commercially available dry films have been investigated but either the spatial resolution is not adequate, the processing time is long, the cost is elevated or the geometrical manufacturing possibilities are limited [5]. For this reason, a novel manufacturing process based on the lamination of successive dry film (DF) photoresist layers (DF-1000 series commercialized by EMS) combined with standard photolithography processes was developed at the Laboratoire d'Analyse et d'Architecture des Systèmes - Toulouse (LAAS) for the fabrication of 3D microfluidic structures in a cost and time efficient manner [5]. This dry film is a negative epoxy about 10 times cheaper than the widely used SU-8, while the processing time for each layer (around 2 h) is 3 times faster than for SU-8. Additionally, the low thermal conductivity of this material (around 1 W m^{-1} K^{-1}) makes it ideal for applications in which thermal management is necessary. The results obtained with this manufacturing process were encouraging and consequently, it has been explored for the fabrication of a 3D gas microfluidic device such as a recently proposed Knudsen pump design [6].

The Knudsen pump is a motionless gas pump, based on the thermal transpiration phenomenon. This kind of pump is able to generate a macroscopic gas flow by applying only a temperature gradient along the walls of a microchannel without any moving parts or external pressure gradient. The design proposed in [6] consists of a series of small and large microchannels drilled across the thickness of a substrate (not etched along the substrate), connecting two reservoirs at different uniform temperatures located at the top and bottom sides of the device. The arrangement of multiple small parallel microchannels leading the pumping effort combined with an adequate number of stages connected by the large microchannels facilitates the development of tailor-made devices for specific performance applications. The proposed channel arrangement of the design provides a compact structure with the cold and hot sides of the device spatially separated, facilitating the overall temperature control. For this reason, the DF manufacturing process developed at LAAS seems a very suitable choice for the manufacturing of these devices, benefitting additionally from the low thermal conductivity of the material.

The manufacturing process for this particular Knudsen pump design requires the lamination and patterning of four different DF layers: first for the bottom reservoirs, second for the microchannels, third for the top reservoirs and fourth for sealing the device with inlet and outlet ports. In this process the critical layer is the second one, in which small microchannels that drive the thermal transpiration flow and larger channels that connect the successive stages of the pump are developed. A scheme of the manufacturing process is presented in Fig. 1 and can be summarized as follows: (i) the DF photoresist layer (uncrosslinked DF) is laminated onto a planar substrate (silicon wafer); (ii) the DF layer is exposed to UV light and baked to catalyze the polymerization reaction following standard photolithography processes; (iii) the DF layer is developed (reticulated DF) in a bath of solvent (cyclohexanone) that removes the material of the non-exposed areas during the photolithography process. This process is repeated for each of the successive layers, enabling the production of different patterns with an alignment precision between the layers of 1 µm. Also, since the typical thicknesses of the commercialized DF layers are 5, 25, 50 and 100 µm (DF-1005, DF-1025, DF-1050 and DF-1100 respectively), different layers can be stacked for a specific pattern, to increase and adjust the thickness of that particular pattern (i.e., the length of the channels) in the device. The best resolution that can be obtained with this procedure depends on the thickness of the DF layer. Using the standard process developed at LAAS, an aspect ratio of 7:1 for free standing structures has been demonstrated, while for channel structures the aspect ratio is 5:1 [5]. Following this process, small circular microchannels with diameter of 10 µm should be possible for a second layer of 50 µm thickness, while the reservoir structures with a resolution of about 20 µm should be achievable within a DF layer of 100 µm for the first and third layers. Large microchannels of 100 µm in the second layer should be easily accomplished for any thickness of the DF layer.

During the first manufacturing attempt, the structures with 20 µm resolution of the first layer were easily and accurately achieved. However, the 10 µm and the 100 µm microchannels of the second layer were not properly developed, denoting an over exposure of the DF. Therefore, the investigation has been focused on achieving the targeted resolution of 10 µm in the second layer for DF layers of 50, 25 and 5 µm thickness. In Tab. 1 the main parameters of the standard process previously developed at LAAS are presented along with the final results from the present investigation. The parameters from the standard process worked properly for the first layer but not for the second layer, resulting in the over polymerization of the 10 µm microchannels. In fact, even when using the standard process for a second layer of 25 or 5 µm, the polymerization of the DF was still excessive and the channels were not completely developed even though the aspect ratio was lower. To reduce the polymerization, the exposure dose and the post-exposure baking (PEB) time have been severely reduced.

First, the minimum exposure dose required to polymerize the interface between the first layer and the second layer has been experimentally identified. When the dose is lower than this threshold, the bottom of the second layer is not properly polymerized and thus, it does not properly bond with the first layer, resulting in the detachment of the second layer during the development step. It is noted that this threshold exposure dose has been approximately determined in this investigation and it is roughly 50% of the one recommended in the standard process. Then, by adjusting the exposure dose (and the associated PEB time) in the range between the standard process and the threshold dose, the development of the 10 µm microchannels has been investigated.

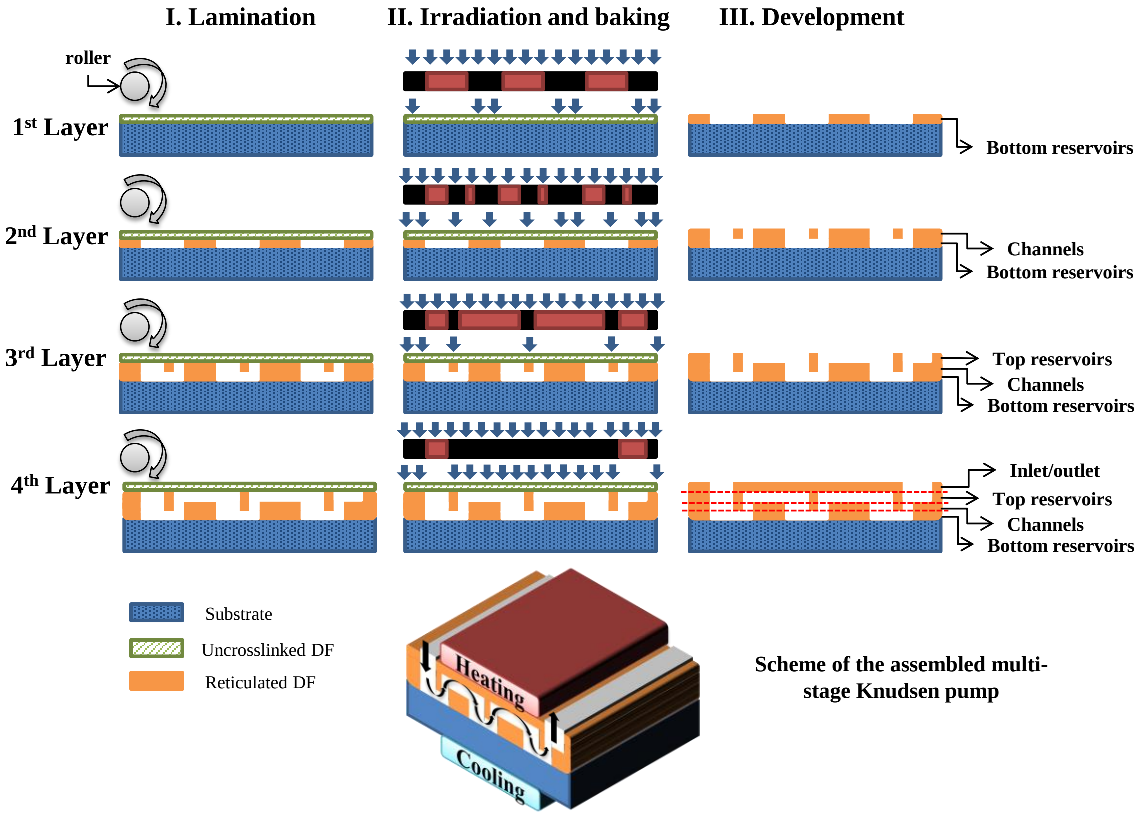

Figure 1: Description of the manufacturing process based on the lamination of DF layers combined with standard photolithography for the fabrication of a Knudsen pump.

		Standard process		Threshold	Improved process for developing 10 µm channels in the second layer	
	Thickness (µm)	Exposure (mJ cm^{-2})	PEB (min)	Exposure (mJ cm^{-2})	Exposure (mJ cm^{-2})	PEB (min)
DF-1100	100	500	10	-	-	-
DF-1050	50	240	6	120	150(*)	4(*)
DF-1025	25	200	5	100	120	3.5
DF-1005	5	160	3	80	100	2.5

Table 1: Summary of the parameters of the standard process developed at LAAS along with the identified threshold exposure dose and the adjusted parameters to achieve a 10 µm resolution in the second layer of any process. For the DF layer of 50 µm the channels with the exposure and PEB denoted by (*) have not completely developed.

For the DF layer of 5 µm (DF-1005) the development of the microchannels has been easily achieved by applying an exposure dose of 100 mJ cm^{-2} and PEB time of 2.5 min. However, due to the tiny thickness of the layer, some areas of the second layer were frequently damaged during the process rendering the sample inadequate. For the DF layer of 25 µm (DF-1025) the targeted resolution of 10 µm has been achieved by applying an exposure dose of 120 mJ cm^{-2} and PEB time of 3.5 min, as shown in Fig. 2a, where multiple parallel channels fabricated across the whole substrate can be observed. Unfortunately, for the DF layer of 50 µm (DF-1050), the 10 µm channels have not been completely developed regardless of the exposure dose of and the applied PEB time. The best results have been obtained with an exposure dose of 150 mJ cm^{-2} and PEB of 4 min. A tentative physical explanation for the over exposure of the second layer is proposed in Fig. 2b. The light is refracted when traversing the deformed areas of the second layer over the cavities formed by the first layer, and it is reflected on the silicon substrate and the walls of the first layer towards the bottom area of the second layer (area not to be exposed) from inside of the cavity. This explains that while the 10 µm microchannels start to develop on the top side, since the bottom part has been over exposed and consequently polymerized, only some tenths of microns are etched on the top surface of the second layer.

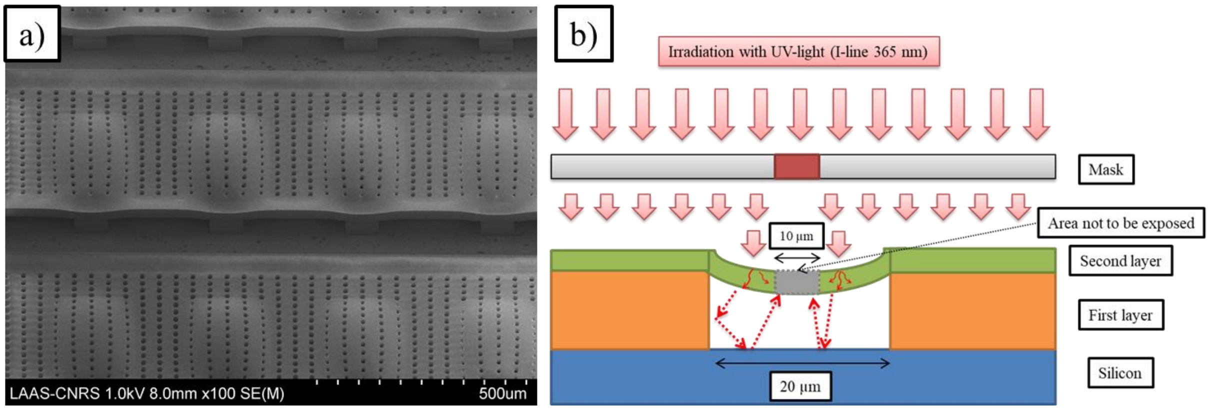

Figure 2: a) Scanning electron microscope image of a sample showing the DF 25 µm second layer displaying the 10 µm microchannels across the whole thickness of the layer and b) proposed physical explanation for the over exposure of the second layer due to the refraction and reflection of the light inside the cavity formed by the first layer.

In conclusion, a manufacturing process based on the lamination of DF layers combined with standard photolithography processes to fabricate complex microfluidic devices has been presented and has been applied for the manufacturing of a particular Knudsen pump comprising four different layers. In this design, the second layer with microchannels of 10 µm is the most important one. The standard LAAS process has been able to generate good first layers but failed to deliver the expected resolution on the second layer due to over polymerization. The threshold exposure dose to start polymerizing the interface between layers and the appropriate parameters to obtain the targeted 10 µm resolution have been investigated. The obtained results have been satisfactory for second layers of 5 and 25 µm thickness, while for the DF of 50 µm further investigation is required to obtain proper microchannels. Finally, a physical mechanism based on the refraction and reflection of light in the microfabricated structure has been proposed to explain the over exposure phenomenon.

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