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The Fabrication of PDMS mould for Microelectrode Array Biochip using NIL

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Abstract. In recent years, low-cost micro and nano fabrication process have gain intention from the manufacturing industry. Biochip is a platform of miniaturized microarrays arranged on a solid substrate that allows various biological tests to achieve immediate results. The development of biochip has established a new platform in biomedical industry. However, to fulfill the demands and availability in the market with affordable cost requires high volume manufacturing techniques for the fabrication of the biochips. In this article we will discuss the fabrication of PDMS mould for replicating microelectrode array of biochip. The fabrication of the microelectrodes utilizes the Nanoimprint lithography (NIL) technique. Finally, the fabrication of PDMS mould has been demonstrated successfully for using Nanoimprint lithography (NIL) technique and achieved 13 % of size difference in overall.

1. Introduction

Biochip is a platform of miniaturized microarrays arranged on a solid substrate that permits multiple tests to be performed at a time in order to achieve quick to responds [1]. Its surface area is no larger than a fingernail [2]. Biochip can perform thousands of biological reactions, such as sorting, trapping, and screening large numbers of biological samples of a variety of purposes, from disease diagnosis, to detection of bioterrorism agents in short time [3]. Lab-on-a-chip (LOC) devices are emerged from the Biochip technology, LOC are broadly used for research in life sciences and diagnostics and represent a very fast moving field [4].

Nanoimprint lithography (NIL) was introduced by Prof. S.Y.Chou and his team in developing a low cost and high throughput manufacturing method [5]. Nanoimprint lithography is a method of fabricating nanometer scale patterns. In the NIL process, it creates patterns by mechanical deformation of imprint resist and subsequent processes. The imprint resist is typically a monomer or polymer formulation that is cured by heat or UV light during the imprinting [6]. Nanoimprint lithography (NIL) is a promising method of high resolution, high throughput and low cost patterning

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technique. The NIL process consist a mechanical replication process where surface reliefs from the template are embossed with a thin layer on the substrate [7].

Poly (dimethyl-siloxane) (PDMS) is a promising material for many applications nowadays because of its outstanding properties. PDMS has been widely used to fabricate Micro-Electro-Mechanical Systems (MEMS), microfluidic dices, micro-stamps by moulding techniques. The PDMS mould will be attached on the imprint roller of roll-to-roll nanoimprint lithography (R2R-NIL) machine for mass production application.

This work will focus on the design and development of PDMS mould of the microelectrode array biochip process.

2. Methods

This section discusses the overall process in developing PDMS mould for microelectrode array biochip, which contains six steps as shown in figure 1. Firstly, the CAD drawn image was plotted on a PET film using image setter technique. The plotted PET film is denoted as master mask film, used in developing a patterned emulsion mask. This was achieved using photolithography technique, since an emulsion mask is a piece of quartz coated with a light sensitive (typically silver halide) material. In step 3, figure 1, contact lithography technique was used to create the SU-8 mould. Then, PDMS (Sylgard® 184 by Dow Corning) was prepared with the mixture ratio of 10 (Base): 1 (Curing Agent) and poured onto SU-8 mould surface. The PDMS thus replicates the inverted mould's profile. Finally, the PDMS mould was slowly peeled off after hard baked in the oven.

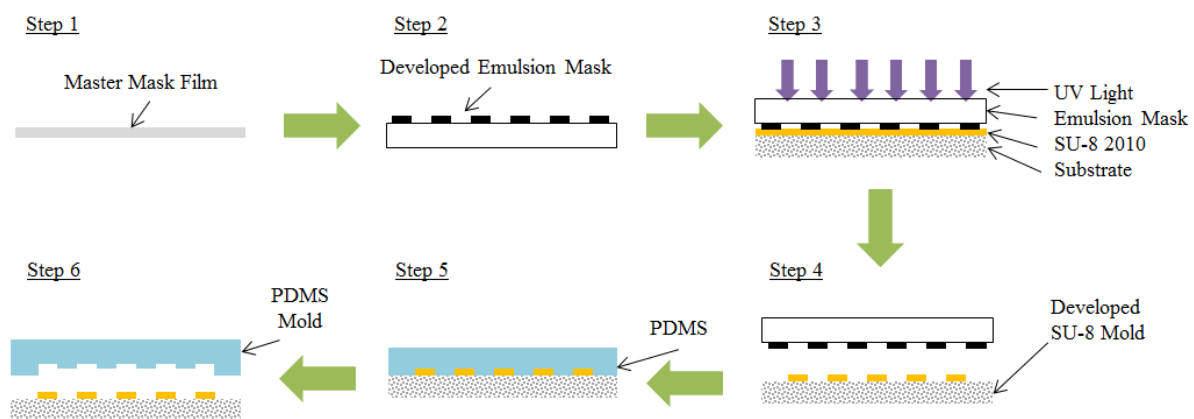


Figure 1. The overall process steps to fabricate PDMS mould.

2.1. Master mask film preparation

The master mask film is essential in fabricating patterned PDMS mould. One of the designs proposed by S.T. Tu et al. [8] was adopted and redrawn using AutoCad 2015 and transferred to CorelDraw. The resulting pattern is shown in figure 2. The pattern was further duplicated in arrays, sufficient to encompass a maximum dimension drawing for the master mask film is up to 210 x 297 mm² (A4 Size).

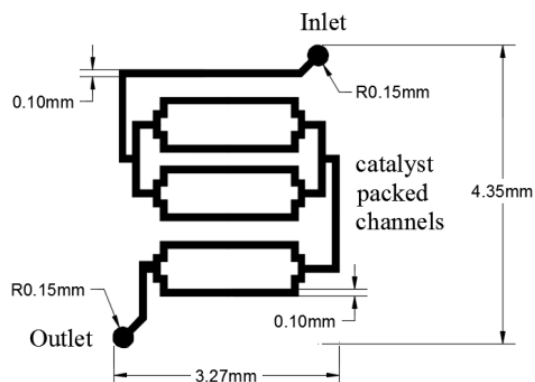


Figure 2. Biochip design pattern sample 3 (Microchannel reactor for gas-phase partial oxidation of toluene), redrawn in AutoCAD 2015 Software [8].

2.2. Emulsion mask development

The simple mask fabrication machine (MM605, Nanometric Technology Inc.) was used to create the patterned emulsion mask through exposure process, as shown in figure 3. Since blank emulsion mask was coated with a light sensitive silver halide coating, the developing process have to be carried out in a dark room. The blank emulsion mask mask (High Precision Photo Plate manufactured by Konica Minolta, Inc.) was placed on top of the mask holder, while that of master mask film on top of a light box. The exposure time was set to 8 seconds, and can be controlled by the shutter door controller. During exposure, light from the light box illuminates the pattern, then projecting it through the lens, scaling down the image to 5 times than its original size.

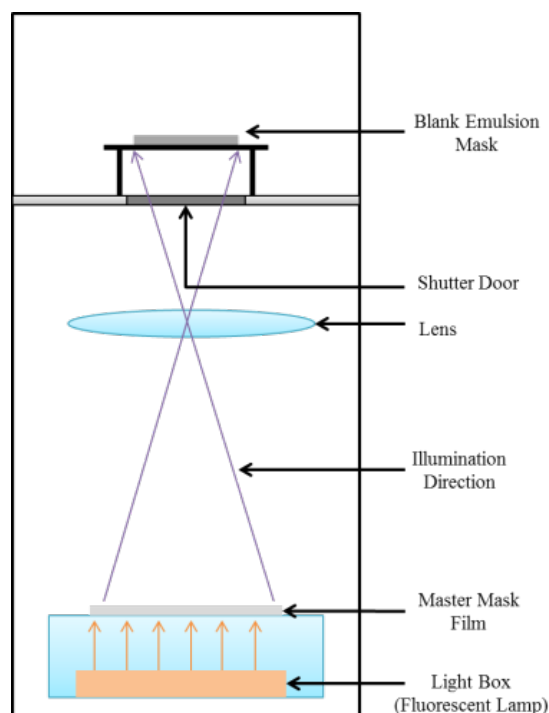
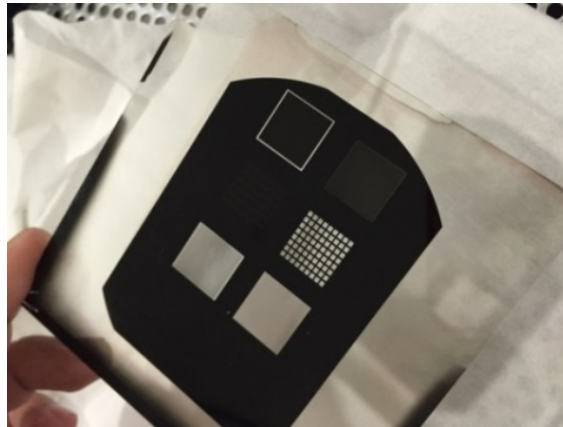


Figure 3. MM605 exposure process.

Table 1. Emulsion mask development steps

	Materials	Immerse time (minute)	Remarks
Step 1	Emulsion Mask Developer (CDH-100) from Konica Minolta, Inc. and Distilled Water	2	Mixture of 1 (CDH-100): 4 (Distilled Water)
Step 2	Distilled Water	2	
Step 3	Fixer Agent (CFL-881) from Konica Minolta, Inc.	10	

The subsequent steps, including the materials used after the emulsion mask exposure were shown in Table 1. The exposed mask was immersed into the developer and stirred for 2 minutes. Followed by distilled water and fixer agent. The developed emulsion mask is shown in figure 4.

**Figure 4.** The developed emulsion mask (4 x 4 Inch²).

2.3. Substrate preparation

Substrate was required to develop a SU-8 mould. The substrate could be silicon wafer, glass or PET film. In this work, a p-type Si wafer (4-inch in diameter with 0.5 mm thickness) served as the substrate and was cut into size of (20 x 20 mm²). Table 2 shows the procedure for substrate cleaning.

Table 2. Substrate cleaning procedures

	Materials	Ultrasonic Cleaning Time (minute)
Step 1	Acetone	5
Step 2	Methanol	5
Step 3	Propanol	5

All the substrate was placed in a beaker with cleaning solvents then placed into the ultrasonic bath for 5 minutes. Then the substrate was blown dry was performed with nitrogen gas and baked on a hot plate for 2 minutes at a temperature of 65 °C, followed by 3 minutes at a temperature of 95 °C.

2.4. SU-8 mould develop preparation process

SU-8 is a permanent epoxy resin formulated for microelectronics and other application which consists of a chemically and thermally stable image. Since SU-8 is highly transparent in the UV region, the

PDMS mould development was recommended. SU-8 can be dispensed with spin or spray methods. SU-8 2010 was selected with spin coating technique. As shown in figure 5, the parameters show the requirement spin speed for SU-8 2010. The spin speed was set to 3500 rpm for 10 μm thickness on the substrate.

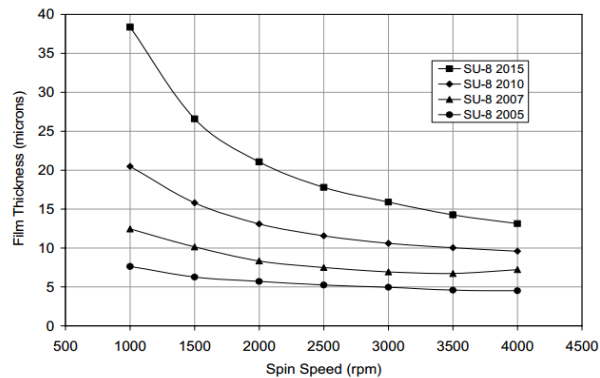


Figure 5. SU-8 spin speed (rpm) vs. thickness [9].

After the spin coating process, the substrate was baked on a hotplate for 2 minutes at a temperature of 65 $^{\circ}\text{C}$, followed by 3 minutes at a temperature of 95 $^{\circ}\text{C}$.

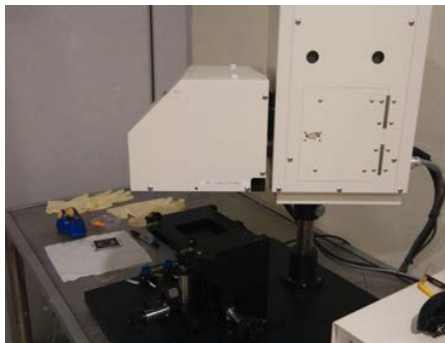


Figure 6. One side mask aligner LA4100_R1 (Sanei Electric Inc.).

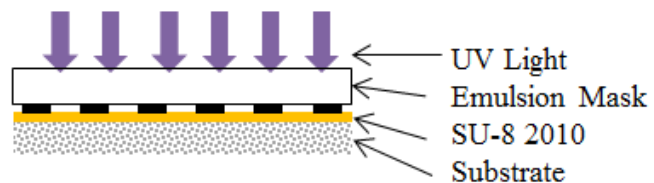


Figure 7. The schematic of exposure setting.

Based on the study of pattern transfer from emulsion mask onto substrate, the emulsion mask and substrate were aligned on the mask holder as shown in figure 6. The ultraviolet exposure system consists of vacuum lock to align both photo plate and substrate in contact. The exposure time was set to 10 seconds. As shown in figure 7, SU-8 layer was exposed when UV light from a source travels through the mask and to the resist. The UV exposure process induces cross-linking of polymer chain in the SU-8 photoresist. The cross-linking process will occur at area that were unprotected by the emulsion mask. After UV exposure, the substrate was post baked on a hot plate for 2 minutes at a temperature of 65 $^{\circ}\text{C}$, followed by 3 minutes at a temperature of 95 $^{\circ}\text{C}$. After post exposure baked, the substrate was allowed to cool down to ambient temperature to prevent deformation.

2.5. SU-8 mould develop process

During the developing process, the unexposed SU-8 photoresist was dissolved by the SU-8 developer (MicroChem). The cross-linked SU-8 photoresist will remained on the substrate. After developing process, the developed substrate was then rinsed by using IPA followed by distilled water. Finally the developed sample was blown dry with nitrogen gas. Table 3 shows the procedure required to develop SU-8 mould.

Table 3. SU-8 mould develop procedure

	Materials	Immerse Time (minute)
Step 1	SU-8 developer solution	1
Step 2	IPA (Isopropyl alcohol)	1
Step 3	Distilled Water	1

2.6. PDMS mould replication process

Sylgard® 184 from Dow Coning was used as Polydimethylsiloxane (PDMS) material. It contains two parts: Base and Curing Agent. The mixture ratio was 10 (Base) : 1 (Curing Agent) [10]. The mixture was stirred for 5 minutes.



Figure 8. Mixture of PDMS on SU-8 mould.



Figure 9. PDMS mould was removed from SU-8 mould.

The mixture of PDMS was then placed in the vacuum chamber for de-gasing process. After that, the PDMS mixture was casted on the substrate surface as shown in figure 8. The substrate with PDMS mixture was baked in the oven at a temperature of 120 °C for 1 hour [11]. After baked, the PDMS mould was allowed to cool down until ambient temperature. The casted PDMS layer was then peeled off slowly by using a razor precision blade to avoid damaging the PDMS mould's structure as shown in figure 9.

3. Results and discussion

3.1. Master mask film and emulsion mask

In order to evaluate the dimension quality of the master mask film and emulsion mask, a Universal Serial Bus (USB) Microscope was used for measurements as shown in figure 10 and figure 11. The measured results were displayed in Table 4. The design of sample 1 was proposed by Samsuri F. et al.[1] while the design of sample 2 was proposed by G.T. Vladislavljević et al. [12].

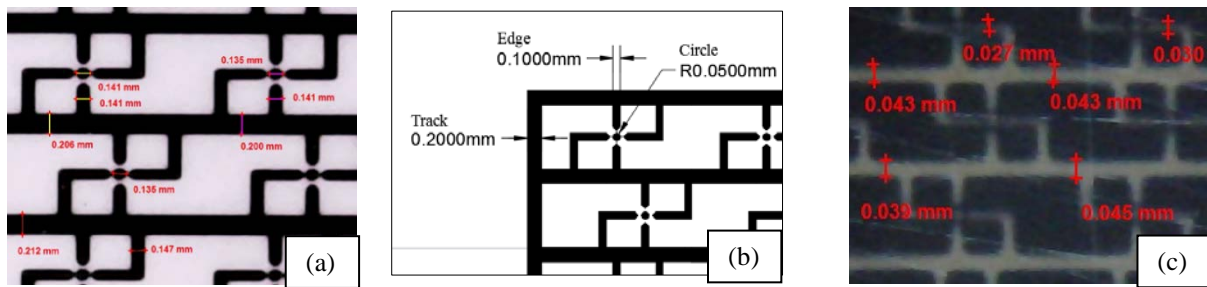


Figure 10. Sample 1: (a) Master mask film, (b) CAD drawing parameters and (c) emulsion mask.

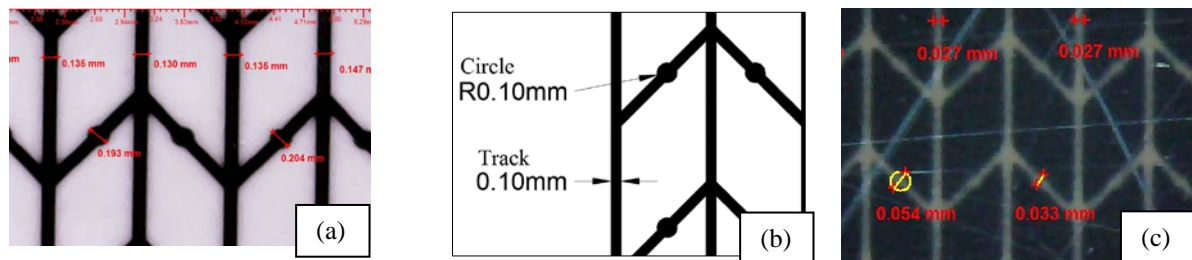


Figure 11. Sample 2: (a) Master mask film, (b) CAD drawing parameters and (c) emulsion mask.

From the USB Microscope measurement results, it was observed that a minor difference between the CAD drawing in figure 10 (a and c) and figure 11 (a and c). Several factors were contributed to this matter; one if it was the master mask film had some slight mis-alignment during the plotting process. A small amount of scratches were observed on the surface of emulsion mask as shown in figure 10 (c) and figure 11 (c). These scratches were caused by the mishandling of the emulsion mask when attaching it on the jig for contact lithography process. Based on Table 4, the results for master mask film shows a greater difference compared to emulsion mask dimension. There were 3 different measurement involved in sample 1 which were circle, edge and track while sample 2 involved only 2 parameters which were circle and track. The edges of sample 1 and 2 have a greater difference compared to circle for sample 1 and sample 2.

Table 4. Measurement of sample 1 and sample 2

	Sample 1 (mm)			Sample 2 (mm)	
	Circle	Edge	Track	Circle	Track
CAD drawing (Actual Size)	0.1	0.1	0.2	0.2	0.1
USB Microscope measurement (Master mask film)	0.1364	0.141	0.206	0.1928	0.1374
Difference between Actual Size and USB Microscope Measurement	36%	41%	3%	-4%	37%
After Exposure Process from Actual Size (5 times smaller)	0.02	0.02	0.04	0.04	0.02
USB Microscope measurement (Emulsion Mask)	0.0246	0.0288	0.0424	0.0318	0.0266
Difference	23%	44%	6%	-21%	33%

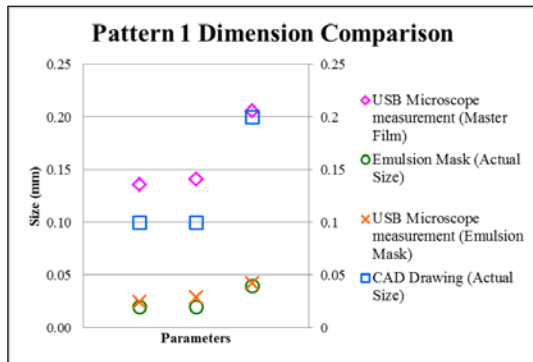


Figure 12. Pattern 1, size (mm) vs. parameters.

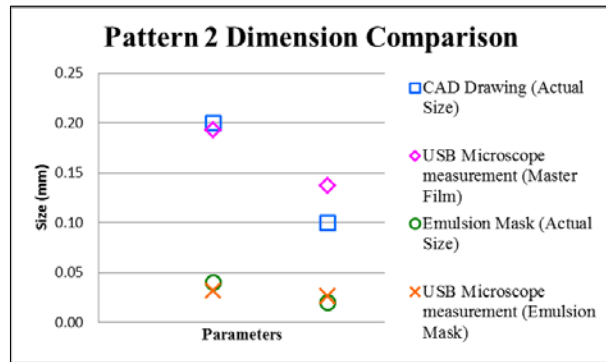


Figure 13. Pattern 2, size (mm) vs. parameter.

Figure 12 and figure 13 illustrate both pattern differences between the actual parameters with the measurement of USB Microscope.

3.2. Sample result comparison between measurement instruments

The measurement performance of USB Microscope was evaluated by comparing a higher resolution microscope, Tukon 1202 where it featured clearer images compared to the USB Microscope as shown in figure 14 and figure 15. Based on the image quality taken from both instruments, the images captured from USB Microscope has a lower resolution compared to the images captured from Tukon 1202. As a result, Tukon 1202 provides a higher resolution and vivid images as shown in figure 14 (a) and figure 15 (a).

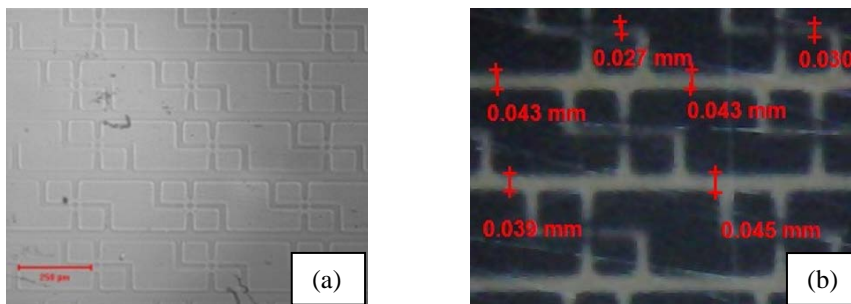


Figure 14. Sample 1 emulsion mask: (a) image captured from Tukon 1202 and (b) image captured from USB Microscope.

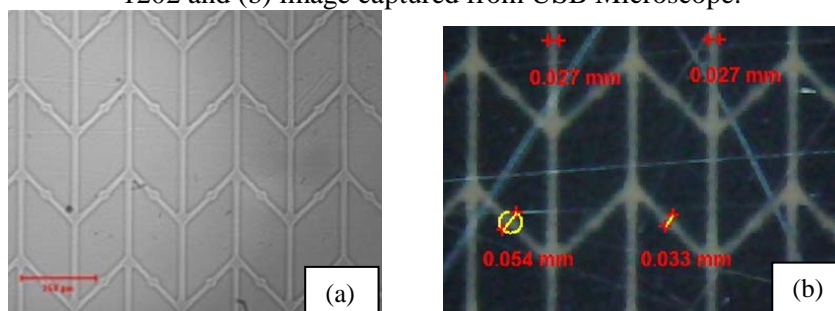
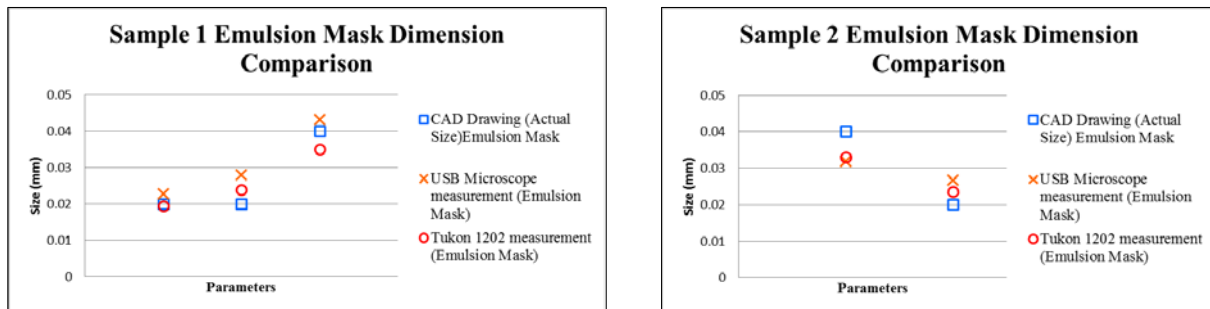


Figure 15. Sample 2 emulsion mask: (a) image captured from Tukon 1202 and (b) image captured from USB Microscope.

Table 5. Measurement between USB Microscope and Tukon 1202

	Circle	Sample 1 (mm) Edge	Track	Sample 2 (mm) Circle	Track
CAD Drawing (Actual Size)	0.0200	0.0200	0.0400	0.0400	0.0200
USB Microscope	0.0228	0.0280	0.0430	0.0318	0.0266
Tukon 1202	0.0195	0.0238	0.0350	0.0330	0.0235

**Figure 16.** Sample 1 and sample 2: size (mm) vs. parameters.

Based on table 5 and figure 16, the results show significantly clearer images because Tukon 1202 has provided more accurate results compared to USB Microscope. The symbols shown in figure 16 shows that the Tukon 1202 was closed to the CAD drawing dimensions.

3.3. Results comparison between different substrates

The Tukon 1202 evaluation of the imprint samples shows satisfied replication for all fabrications without major defect issues.

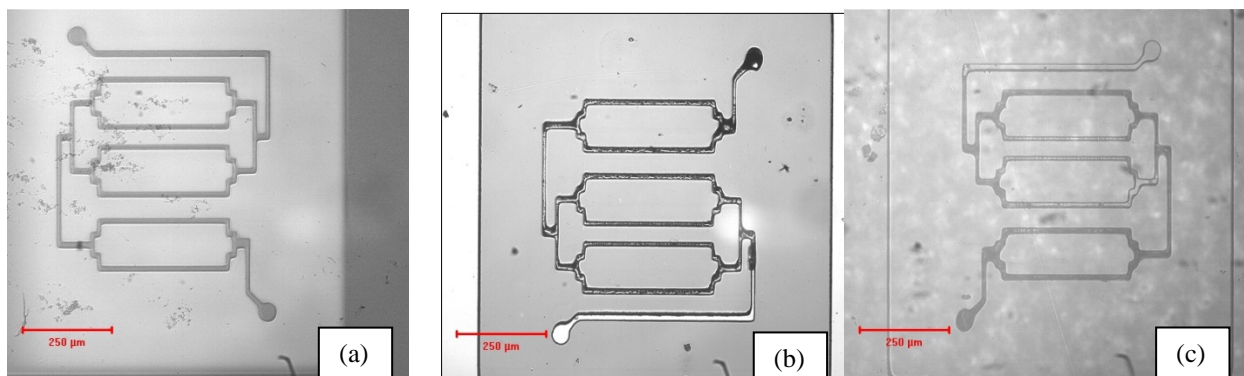
**Figure 17.** Replication sample on: (a) emulsion mask, (b) SU-8 mould and (c) PDMS mould.

Table 6. Measurement of all substrates replication

	Emulsion Mask (μm)		SU-8 Mould (μm)		PDMS Mould (μm)	
	Inlet/ Outlet	Channel's Width	Inlet/ Outlet	Channel's Width	Inlet/ Outlet	Channel's Width
Actual	60.0000	20.0000	60.0000	20.0000	60.0000	20.0000
Overall	65.9338	17.2535	73.7834	17.4108	58.3146	16.9720
Difference	10%	-14%	23%	-13%	-3%	-15%

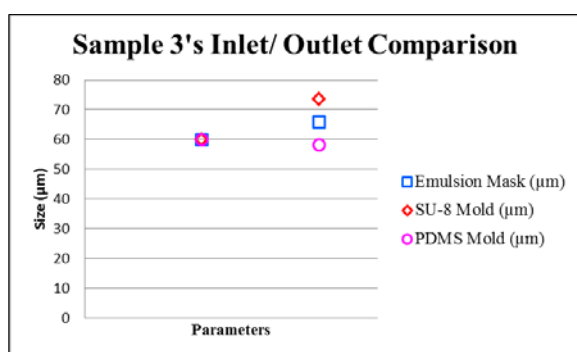
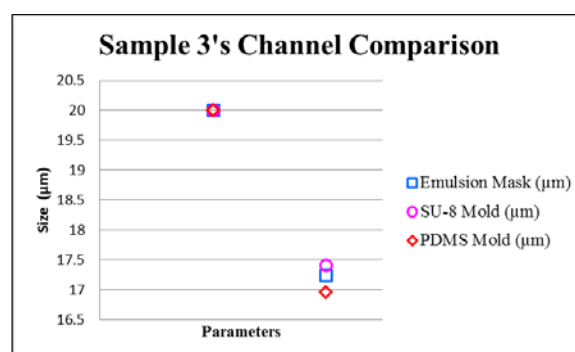
**Figure 20.** Sample 3's inlet/ outlet comparison.**Figure 21.** Sample 3's channel comparison.

Figure 17 shows the replication pattern of a Microchannel reactor for gas-phase partial oxidation of toluene designed by S.T. Tu et al[8]. Based on the measurement performed, the difference value of positive indicates the replication size is larger from the original size. However, the replication size that carries negative values is smaller from the original size. The measurement results for all the fabricated samples were shown on Table 6 along with figure 20 and figure 21. There were 2 parameters applied in this measurement which are circle and track. The circle was located on the top and bottom area of this sample and track was the width of the microchannel.

4. Conclusion

The accomplishment of this work has provided a potential solution towards low cost and high yield manufacturing process for the fabrication of the biochip. The overall results showed satisfied results upon all the development from master mask film until PDMS mould. The UV exposure time was one of the important elements for replication technique. Using Tukon 1202 provides higher image quality compared to USB Microscope at least 30%. The results of Tukon 1202 show that all fabrication's dimensions were actually have 13% differences from the actual dimension. This has proved that the PDMS mould is capable to be fabricated using nanoimprint lithography technique. The PDMS mould then can be fixed onto the imprint roller of roll-to-roll nanoimprint lithography (R2R-NIL) machine.

5. Acknowledgement

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