MICROSTRUCTURING OF SU-8 RESIST FOR MEMS AND BIO-APPLICATIONS

P.K. Dey[1], B. Pramanick[1], A. RaviShankar[2], P. Ganguly[1], and S. Das[3]

[1]Advanced Technology Development Centre

[3]School of Medical Science and Technology

Indian Institute of Technology, Kharagpur-721 302, India.

[2]Department of Electronics and Communication Engineering,

School of Electrical Sciences, Karunya University, India.

Email: pkd@ece.iitkgp.ernet.in

Abstract- Some studies on the fabrication of micro-needles, micro-pillers, and micro-channels using SU-8 negative photoresist for MEMS and bio-applications are reported. The SU-8 processing technology was standardized for the purpose. Micro-pillars were fabricated on SU-8 polymer by soft lithographic technique. Micro-needles were realized on SU-8 film utilizing lensing effect of the etched groove structure of the glass substrate. Micro-channel was fabricated by molding of PDMS polymer on patterned SU-8 ridge structure. Structural characterization of the fabricated structures were investigated using optical microscope and SEM.

I. INTRODUCTION

With the advancement of micromachining technology various miniature transducers made from silicon, glass and polymer materials are becoming more popular for its potential applications in biological and medical fields. Microfluidic devices, micro-needles and micro-pillars made from polymer materials using micromachining technology are regularly used in drug delivery system, biological processes in microenvironment, cell manipulation, etc. Commonly available needles are effective at bolus delivery of drugs, but cause pain during insertion and are not ideally suited for delivery over extended periods. Transdermal patches address these shortcomings, but are extremely limited in application because most drugs are unable to cross skin at therapeutic rates. As a novel alternative, micro-needles fabricated by micro-fabrication technology are used [1] that pierce into the skin far enough to permit drug delivery, but are short and thin enough to avoid causing pain. Similarly the generation and sensation of mechanical force plays a role in many dynamic biological

processes, including touch sensation [2]. Micro-pillar on quartz substrate can be utilized in measuring tactile sensitivity and interaction forces exerted during locomotion by small organisms. In a similar manner micro-channels are regularly used as fluid mixing, cell culture, enzyme reactor [3] and heat exchanger [4]. Polymer based micro-structures are becoming more popular in this field for past few years. In this context SU-8, a chemically amplified, high-contrast, epoxy-based negative tone photoresist is very popular for micromachining and microelectronic applications. It has very high optical transparency above 400 nm, and is ideally suited for patterning high aspect ratio with near vertical sidewalls structure in very thick films. As a photoplastic material, SU-8 is chemically stable and resistant to most acids and other solvents. Consequently, it is difficult to remove once cross-linked. The mechanical, chemical and optical properties and processing parameters of SU-8 have been investigated by various groups [5]. In this paper the SU-8 processing techniques developed in the authors' laboratory for the fabrication of micro-needles, micro-pillars and micro-channels, for micro-system and bio-applications, are described. Although some more process optimization of the process steps and structural modification are required for the intended applications, the initial results are presented.

II. METHODOLOGY

Array of micro-pillars and needles were realised on SU-8 polymer and microfluidic channel was realized on PDMS polymer using patterned SU-8 as a mold. All structures were fabricated on glass substrate. The mask layout was done using CAD software and dark field mask was fabricated for each individual structure using in-house direct laser writing system (Microtech Laser Writer LW-2000).

A. MICRO-PILLAR FABRICATION

Fabrication of micro-pillars of SU-8 polymer was realised on glass substrate. Dark field mask containing array of circular window of varying diameter from 100 µm to 300 µm and separation 150 µm was designed for this purpose. To start the fabrication process the glass substrate were cleaned in $H_2O_2 + H_2SO_4$ (1:1) solution for 20 min and then cleaned in de-ionised water followed by drying using nitrogen gas. SU-8 (MicroChem Corp., SU-8, 2075) negative photoresist was spin coated on the glass substrate and then exposed to ultraviolet (UV) light for transferring the mask pattern on the SU-8 layer. Initially 3-4 ml of SU-8 photoresist was spin coated at 500 rpm for 10

sec, followed by ramped up to 1000 rpm and spun for another 20 sec to obtain 250 µm thick resist layer. Subsequently, the SU-8 coated glass substrate was kept on a perfectly leveled surface for 15 min to allow the resist to acquire uniform thickness. The coated substrate was then soft baked on a hotplate at 65°C for 7 min and ramped to 95°C and baked again for 30 min. The substrate was then allowed to cool down for thermal relaxation for about 10 min. After thermal curing the SU-8 coated glass substrate was exposed to UV light of 405 nm wavelength through the dark field mask. The required exposure time was 45 sec to expose 250 µm thick photoresist. Post exposure baking was done immediately after UV light exposure in order to cross link the film on a hot plate at 65°C for 5 min and then ramped to 95°C and kept for another 12 min. Finally the photoresist layer was developed by immersion technique in a developer solution (MicroChem's SU-8 Developer) for 12 min to release the micro-pillar structure. The process step is shown in fig. 1. Mild ultrasonic agitation was required during development process for proper release of the micro-pillar structure. Since SU-8 is a soft material and the aspect ratio of released pillars are quite high, extreme precousion were taken during development and post exposure bake process to keep the pillars vertically up. After complete polymerisation, the material gets hardened and thus handling becomes easy.
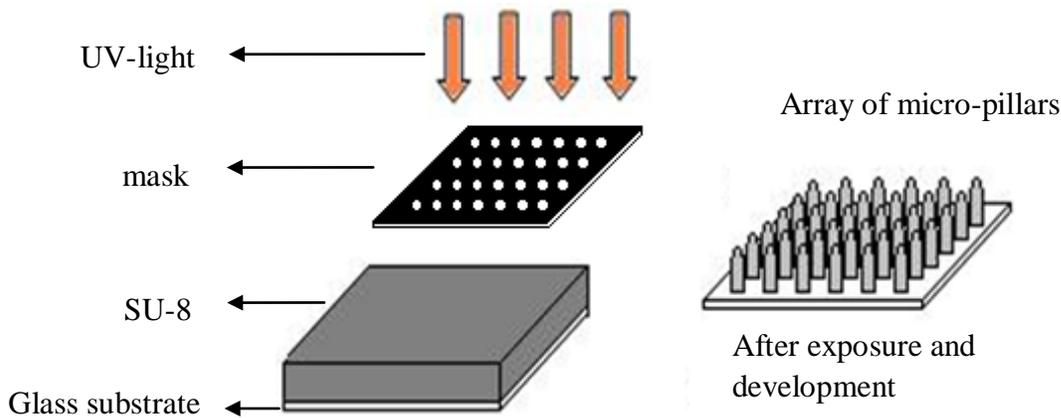

Fig. 1. Schematic of SU-8 micro-pillar fabrication

B. MICRO-NEEDLE FABRICATION USING LENSING TECHNIQUE

Basically the micro-needle is a conical shape structure with a through hole along its axial direction. In the initial attempt fabrication of an array of conical shape structure on SU-8 layer by micromachining technique was aimed. In this context, the semi circular groove made on glass substrate was utilized to focus the exposed UV light in the SU-8 film. This lensing action will tailor

the UV light beam in a 3D conical shape resulting conical shaped exposed area in the SU-8 resist. The realization of micro-needle requires two separate process sequences: a) fabrication of semicircular etched grooves with curved surfaces in glass substrate and (b) fabrication of SU-8 based needles on the grooved glass substrate by using lensing action during exposure. The schematic of complete process sequence is given in fig. 2.

(a) Glass substrate

(b) Deposited Cr-Au layer

(c) Lithography to open Window on Cr-Au layer

(d) Glass Etching

(e) SU-8 coating on grooved surface

(f) UV Exposure from glass side

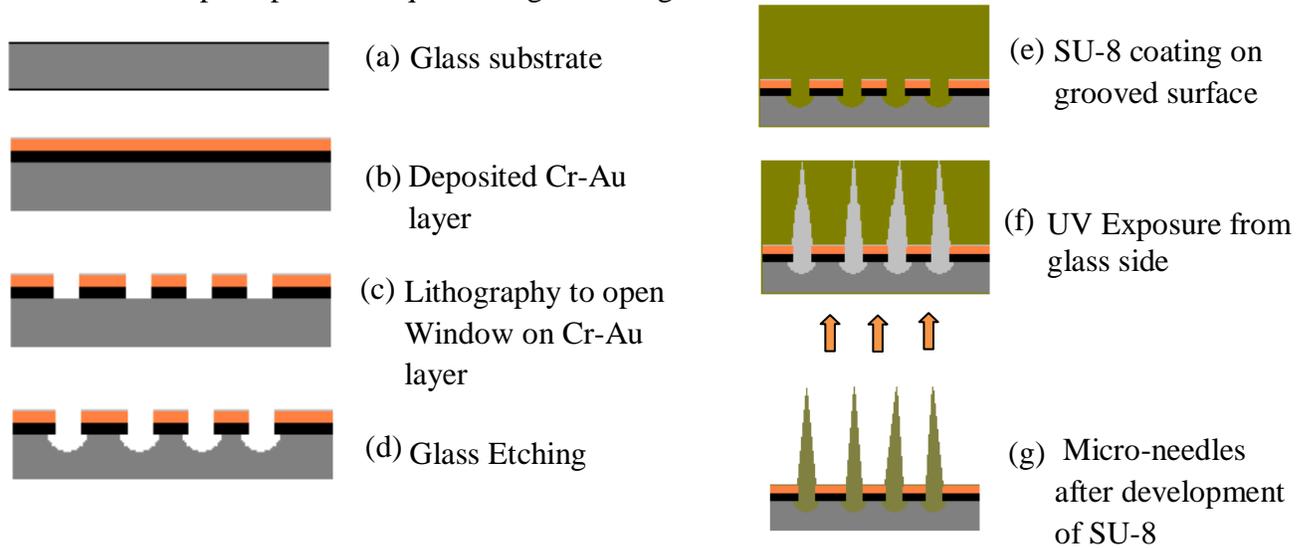(g) Micro-needles after development of SU-8

Fig.2. Process sequence for fabrication of micro-needle.

FABRICATION OF GROOVES ON GLASS SUBSTRATE.

The process to fabricate micro-needle begins with the fabrication of almost spherically etched grooves with circular opening in borosilicate glass substrate. The structure was realized by isotropic etching of glass substrate in a chemical solution through chromium-gold (Cr-Au) masking layer. Initially substrates were cleaned in $H_2O_2 + H_2SO_4$ in 1:1 solution for 20 min. followed by rinsing in DI water and drying . The cleaned glass wafers were then loaded in thermal evaporation system (Hind Hi Vac, India) to deposit Cr-Au thin film layer. Typical deposition pressure was 7×10^{-6} mbar with a substrate heating at $120^{\circ}C$. Thin layer of Cr of about $200 A^0$ was deposited on the glass substrate followed by deposition of Au-layer of thickness 0.3 µm (fig. 2. b). This Cr-Au film was used as a masking layer for the subsequent glass etching process.

Dark-field mask consisting of an array of 50µm, 100 µm, 150 µm and 200 µm diameter circular windows was designed and the mask was fabricated. Conventional photolithography with positive photoresist (Fuji Film: HPR 504) has been adopted to create circular windows in the deposited Cr-Au layer on the glass substrate using the fabricated mask as shown in fig. 2(c) The Cr and Au layers

were etched in their corresponding wet etching solutions (Transene Inc. USA) at room temperature for 1-3 min., as required for the desired thickness to be etched. Finally the positive resist was stripped off completely in warm (40^0 C) acetone for 10-15 min. In the next step the glass substrates were isotropically etched through patterned Cr-Au windows using HF (5 ml) + HCl (10 ml) + DI water (85 ml) solution at room temperature for different durations of time. The etching time was adjusted carefully to achieve the required grooves with smooth curved surfaces of different curvatures in the glass as shown in fig. 2(d). The etching of glass and the etched profile were optimized for the present requirement. The typical etch rate of glass was about 0.4 µm/min at room temp.

FABRICATION OF MICRO-NEEDLE IN SU-8 LAYER

A layer of SU-8 polymer was used to fabricate micro-needles on top of the grooves created in the glass substrate by UV-lensing effect. In this study initially SU-8 photoresist (MicroChem Corp. SU-8 50) was spin coated at 500 rpm for 10 sec, followed by spinning at 1000 rpm for 20 sec to obtain 100 µm thick resist layer. The coated substrate was then kept on a leveled hot plate to acquire uniform thickness and soft baked at 65^oC for 10 min and then to 95^oC for 30 min. After thermal curing, the total structure was illuminated through the glass side by UV light of 405 nm wavelength for 35 sec. as shown in fig. 2(f). In this step convex action will occur while the light beam passed through the etched grooved structure of the glass substrate of refractive index 1.5 to SU-8 layer of refractive index 1.67. By this process UV-light was focused within the thick photoresist layer through the circular masking windows of the Cr-Au layer. Post exposure baking was done as stated in the previous section. A careful development in a developer solution for 8 min, released an array of rigid micro-needles, which actually replicates the focused light-pencil during exposure as shown in fig. 2(g). The proper development of SU-8 has been verified by rinsing in isopropyl alcohol, which otherwise results in precipitation of white colour material on the sample and turns isopropyl alcohol milky solution. The best results have been obtained with a mild ultrasonic agitation during development. The critical parameter to form highly pointed micro-needles is the proper matching of the SU-8 thickness with the focal length (f) of the convex interface between the glass substrate and SU-8 layer of the etched grooves. The focal length (f) is dependent on the radius of curvature (r) of the groove by the relation [6]:

$$f = r \mu_2 / (\mu_2 - \mu_1) \tag{1}$$

where μ_1 (=1.5) and μ_2 (=1.67) are the refractive indices of glass and SU-8 respectively at UV wavelength. Sharply pointed micro-needles are expected for SU-8 thickness equal to the focal length (f). Thus proper combinations of SU-8 thickness, window diameter and radius of curvature of the etched groove structures can be derived from eq.(1) to design micro-needles of different heights.

C. MICRO-CHANNEL FABRICATION

In another experiment, micro-channels of various shapes and geometry made of polydimethyl siloxane (PDMS) were fabricated on the glass substrate by micro molding process. In this case the micro-channel pattern was first fabricated on SU-8 polymer as ridge like structure by lithography process and then it was used as mold to transfer the reverse pattern on the PDMS polymer.
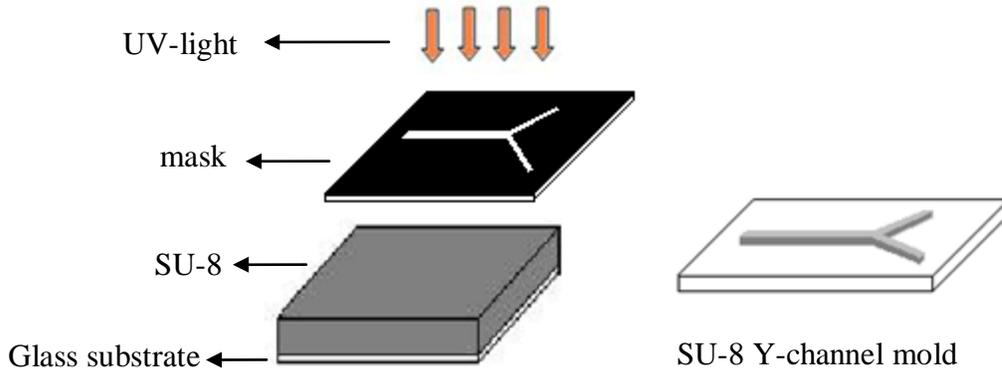
fig. 3. Schematic of the process for realization of SU-8 mold containing ridge shaped micro-channel.

Schematic of the process for realization of the SU-8 ridge structure on glass substrate is shown in fig. 3. A dark field mask of various micro-channel structures were made for different geometrical dimensions. Typically the micro-channel length was 10 mm, width was 100 µm and thickness was 50 µm. To obtain the desired micro-channel height of 50 µm, the SU-8 photoresist was spin coated on glass substrate at 500 rpm for 10 sec and ramped to 2000 rpm for 20 sec. followed by soft baking as mentioned in previous section. The resist was exposed to UV light for 25 sec through dark field mask. Subsequently the film was properly baked and carefully developed for 6 min to obtain the ridge structure of SU-8 layer which was used as mold for realization of PDMS micro-channel structure. In this process a thick PDMS layer was coated on the mold structure. 10:1 ratio of PDMS (Dow Corning Sylgard 184) base and curing agent has been mixed thoroughly and then degassed in

a vacuum chamber for about 20 min. A thick layer of PDMS was spin coated on the SU-8 mold structure and then thermally cured at 80^0 C for 40 min in order to cross link and polymerise the PDMS layer. After cooling down, to room temperature, the PDMS film was carefully peeled off from the mold surface and thereby the reverse pattern of SU-8 structure i.e. an engraved pattern of micro-channel was created on the PDMS layer. The engraved pattern of PDMS layer was then transferred and fixed to a clean glass substrate for complete realization of micro-channel. The bonding between PDMS film and glass substrate was carried out in a conventional oven at 150^0 C for 2 hrs. This process resulted in realization of micro-channel covered with PDMS on glass substrate . The entire PDMS process is shown in fig.4. Finally the input and output holes were made by punching at the extreme points of the micro-channel and fine capillary tube was fixed by glue epoxy for liquid flow [7].
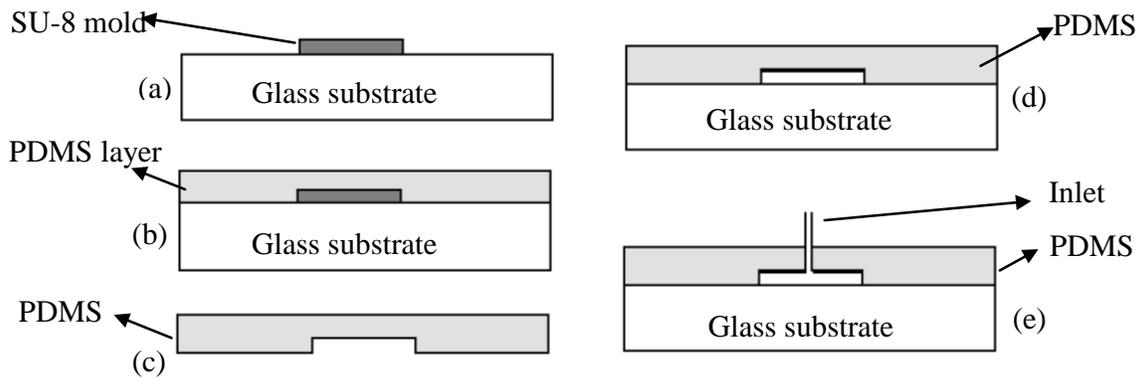

Fig. 4. Process steps for fabrication of micro channel.

III. RESULTS AND DISCUSSIONS

Structural characterization of the fabricated microstructures was investigated using optical microscope and SEM. Fig. 5 shows the optical and SEM photographs of the fabricated array of micro-pillars of SU-8 polymer. It may be observed from the photographs that almost vertical micro-pillars with nearly uniform circular diameter and height were obtained in the microstructure. The measured diameter and height of the pillar were about 180 µm and 250 µm respectively. The uniform circular wall was obtained for almost all pillars in 10x10 array by careful development of photoresist with mild ultrasonic agitation. A special care was taken during processing to keep the released structure stands almost vertical.
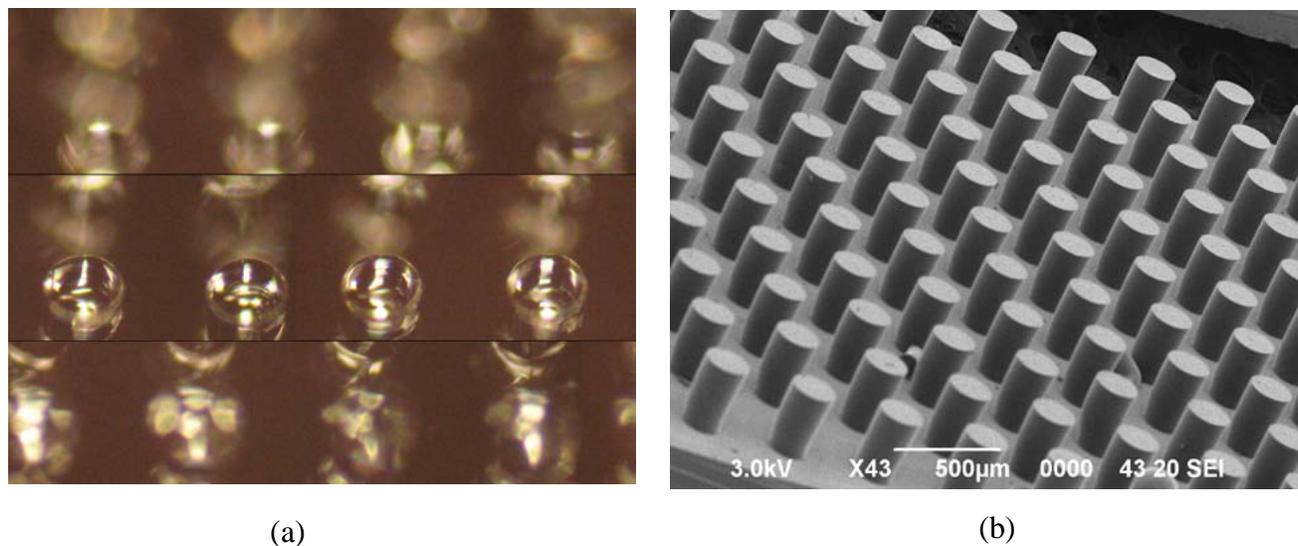
(a)

(b)

Fig.5. (a) Optical and (b) SEM images of an array of micro-pillars.

The critical issue for realizing perfect conical shape micro-needle is to achieve perfect semicircular etched grooves with circular opening on the glass substrate. Thus, the etched grooves made on glass substrate were thoroughly investigated by optical microscope and its cross section etched profile was measured by a surface profiler (Sloan Dektak-3). An optical image of the top view of isotropically etched grooves of different diameters along with Cr-Au masking layer is shown in fig. 6.
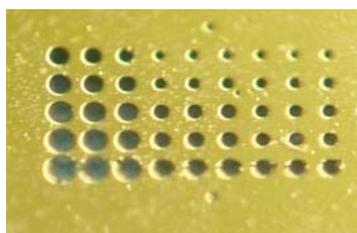
Fig. 6. Optical image of the glass substrate consisting
of 9x5 array of etched grooves of varying diameter.

A typical cross sectional etched profile of a groove of 100 µm diameter circular opening on the glass substrate after 1 hr. etching in HF (5 ml) + HCL (10 ml) + DI solution is shown in fig. 7. The measured etch rate of glass was 0.4 µm/min. Etch depth of the groove was found to be ~ 28.5 µm after 1 hr. etching time as observed in fig. 7. The profile was asymmetric along the lateral direction which is not desirable. This result may be due to error in lithography and defects in the glass

substrate which is not of high quality. There was also noticeable lateral etching due to isotropic characteristics of etching solution. Although the diameter of circular window opening in the Cr-Au mask was 100 µm, the measured diameter after etching was found to be ~ 120 µm as observed from fig.7. The measured lateral vs vertical etch ratio was 0.7. The average surface roughness measured by surface profiler was 183.4 A^0 for the etching solution used in this study.
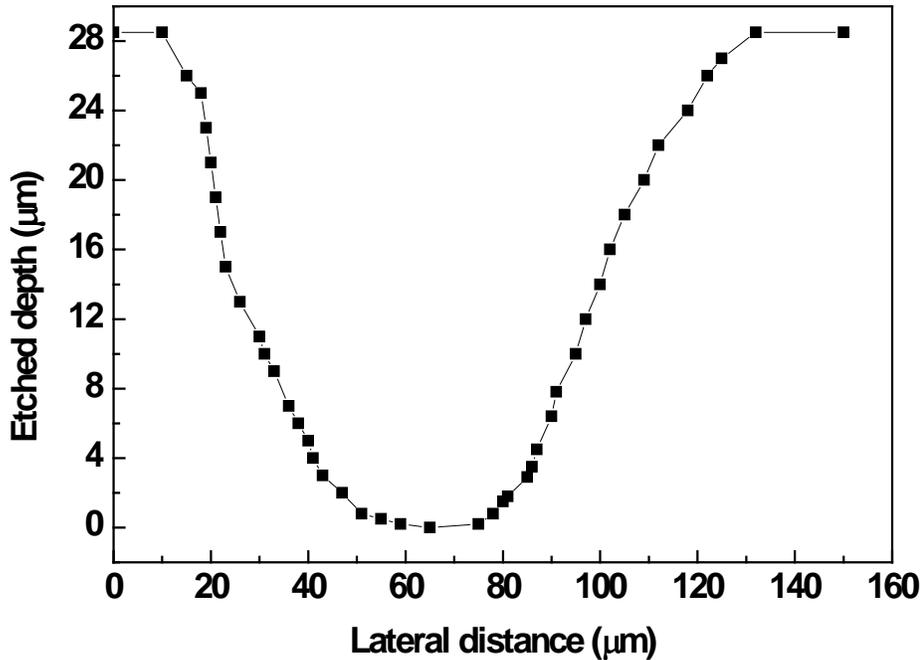

Fig. 7. A typical etch profile of the groove of 100 µm diameter circular opening on glass substrate after wet etching.

The fabrication of sharply pointed micro-needles depends on the careful matching of fabrication parameters, such as resist thickness, curvature of grooves and its depth. In this study, 100 µm thick photoresist layer with more than 50 µm diameter window size were used. However less than 20 µm diameter window size is required to get the sharply pointed needle structure. Optical photograph of the fabricated micro-needle of 50 µm diameter and about 95 µm height with different shape of the tip is shown in fig. 8. Using same SU-8 resist thickness the micro-needle was obtained with comparatively sharp tip as shown in fig 8 (a) using the etched groove of lesser radius of curvature, whereas micro-needle with blunt tip as shown in fig. 8(b) was obtained using larger groove diameter.
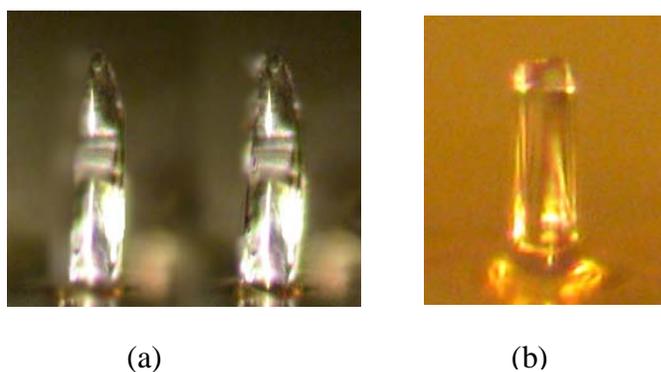
(a)   (b)

Fig.8. Fabricated micro needle: (a) sharp tip and (b) blunt tip.

Micro-channels of various design were fabricated by micro-molding of PDMS polymer using patterened SU-8 structure. Optical photograph of a typical Y shaped SU-8 ridge structure is shown in fig. 9. This structure consisting of two inputs and one output port was chosen to observe the fluidic flow pattern in a micro channel under various pressure driven flow. The fabricated PDMS based micro-channel integrated with input/output ports and inlet tubing connections for fluidic flow is shown in fig. 10. The length, width and height of channel were 10 mm, 100 µm and 50 µm respectively. The lateral dimension were fixed by mask geometry while required height of the channel were controlled by patterned SU-8 thickness. Detailed observations reveals that the width and height were quite uniform along channel length direction and the wall surface was smooth enough for laminar flow pattern.
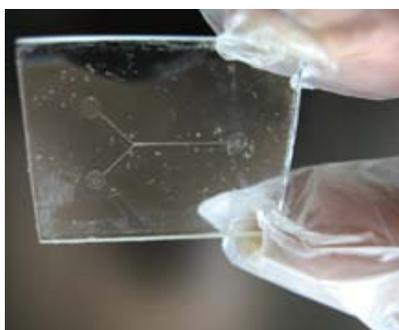

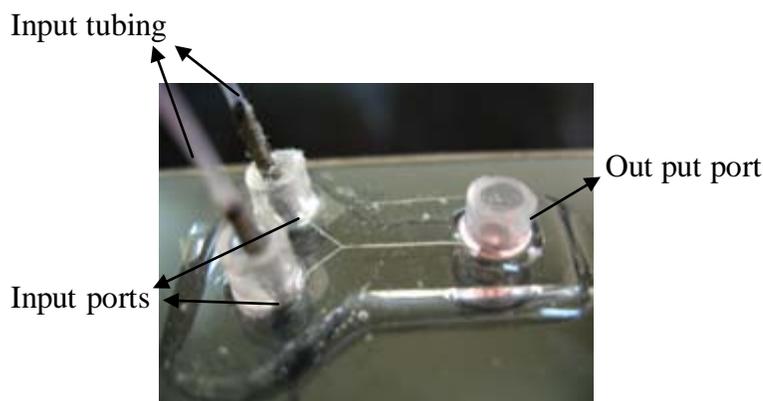Fig. 9. Optical photograph of fabricated pattern as a mold for making Y shaped micro-channel.

Fig. 10. Fabricated Y shaped micro channel using PDMS.

IV. CONCLUSION

An experimental study to fabricate micro-needle, pillars and channels using SU-8 polymer by soft lithographic technique is reported. For this purpose lithographic processes for SU-8 resist of thickness 50 µm to 250 µm were standarised. Array of micro-pillars of diameter 180 µm was successfully realized. Micro-needles were realized on SU-8 polymer by utilizing lensing effect at the curved interface between SU-8 layer and etched glass surface and the difference of refractive indices of the two medium. Y shaped micro-channel was fabricated in PDMS polymer by micromolding process using patterned SU-8 ridge like structure as mold. Although various structures were realized for different applications, further investigations are required for improved and repeatable yield.

ACKNOWLEDGEMENT

REFERENCE

1. J-H Park, M.G. Allen, and M.R. Prausnitr, "Biodegradable polymer microneedles: fabrication, mechanics and transdermal drug delivery", *J. of Controlled Release*, vol. 104, pp. 51-66, 2005.

2. Joseph C. Doll, Nahid Harjee, Nathan Klejwa, Ronald Kwon, Sarah M. Coulthard, Bryan Petzold, Miriam B. Goodman and Beth L. Pruitt, "SU-8 force sensing pillar arrays for biological measurements", *Lab Chip*, vol. 9, pp. 1449 - 1454, DOI: 10.1039/b818622g, 2009

3. Masaya Miyazaki and Hideaki Maeda," Microchannel enzyme reactors and their applications for processing", Trends in Biotechnology, Vol. 24 No. 10, 2006.

4. Seok Pil Jang, Stephen U.S. Choi, "Cooling performance of microchannel heat sink with nanofluids", Applied Thermal Engg. Vol. 26 , pp. 2457-2463, (2006)

5. Lorentz H, DespontM, Fahrni N, LaBlance N, Renaud P "SU-8: a low cost negative resist for MEMS ",J. Micromech.Microeng. pp.7121, 1997

6. D.P. Acharya, "Geometrical Optics for Advanced Students", Oxford & IBH Publishing Co. Pvt. Ltd., New Delhi, 1987.

7. J.C. McDonald, G.M. Whitesides,"Poly (dimethylsiloxane) as a Material for Fabricating Microfluidic Devices," Accounts of Chemical Research, Vol.53 no 7, pp 491-499, July 2002.

8. Lorentz H, Laudon M and Renaud P "Mechanical characterisation of a new high-aspect-ratio near UV resist", Microelectron. Eng.vol. 371, pp. 41-42, 1998

9. Zhang J, Tan K L, Hong G D, Yang L J and Gong H Q"Polymerisation optimisation of SU-8 photoresist and its applications in microfluidic systems and MEMS", J. Micromech.Microeng. vol. 11, pp. 20, 2001