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Invited Lecture

Soft-lithography: Its application in solar cells, microelectronics and life sciences

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The soft-lithography technique and its variants, namely micro contact printing (μCP), micro replica moulding (REM) and micro moulding in capillary etc. have been reviewed. The key elements of soft-lithography: the poly-dimethylsiloxane (PDMS) properties, fabrication sequence, and assembly have been discussed. The review also looks at the efforts that are being made to modify the PDMA surface for bonding, μCP of bio-molecules etc. A of alkanethiols on noble metals is used as a resist against chemical etchants and patterning of thin Au, Ag, Ti and Pd films (features ranging from sub-μm to tens of μm) has been demonstrated on planar and curved surfaces. In addition, an array of contrasting surface properties has been made by sequential formation of self-assembled monolayer (SAM). A Y-shaped channel and a network of ten micro-channels (40-80 x 75 μm^2 cross-section) has been realized in PDMS using REM. Soft-lithography, being a cheap and simple fabrication process with rapid prototyping, offers the option of low cost production of miniaturized devices, and has potential applications in microelectronics, chemical and life sciences.

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1. Introduction

"*Soft-Lithography*" is a generic term for several lithographic techniques, namely micro contact printing (μCP), micro replica moulding (μRM or REM), micro moulding in a capillary (MIMIC), solvent assisted micro-moulding (SAMIM), and micro transfer moulding (μTM) for fabricating micro- and nano-structures [1]. Whitesides and co-workers [2] developed soft-lithography in the early 1990s at Harvard University. The two key elements of soft-lithography technology are (i) a master wherein the desired patterns are created either in silicon (using standard Micro-Electro-Mechanical Systems (MEMS) processes) or in SU8 (a negative photo-resist) and (ii) a stamp of poly-dimethylsiloxane (PDMS) made by a moulding process inside the master, that has the relief structure of the master. Patterns and structures ranging from a few tens of nm to several hundreds of μm can be made using soft-lithography, that would have widespread applications in biological, biochemical, microelectronics, and micro-optical systems.

In μCP, alkanethiols that self assemble on a coinage metal (Au, Ag, Cu, Pd etc) surface to form a dense and ordered monolayer is transferred selectively using an elastomeric stamp. The self-assembled monolayer (SEM) acts as a nano-thick resist against metal etchants, and un-derivatized metal regions can be etched to obtain the desired patterns on the metal surface. μCP is promising in micro-fabrication and patterning of metal (single or multi) layers for electrical contacts. On the other hand, the self assembled monolayer, (SAM) of alkanethiols with different head groups can realize an array of alternating hydrophilic and hydrophobic lines or islands on a metal surface. Such a structured surface has potential applications in biological and biochemical analysis.

Micro-fluidic channels (μFC) and their network (μFN) can be created in PDMS by using REM, the other variant of soft-lithography. In MIMIC (where μFCs are formed by bringing the PDMS mould into conformal contact with a substrate) has been used to pattern a variety of materials such as colloidal particles, polymer beads, inorganic salts, etc. It can be used to pattern biological molecules such as immuno-globulins by allowing solutions to flow through micro-channels that restrict the biological legends at well defined regions on the substrate. Soft-lithography is also usefully employed to fabricate miniaturized functional components such as pumps, mixers, valves and chambers that can handle the flow of nano- to micro-litre solutions. The PDMS based microfluidic systems are easy to fabricate and can be sealed reversibly or irreversibly with other surfaces. Therefore, the system assembly is rather easy. For example, "Lab-on-a-Chip" (LoC) technology comprising of tiny chambers and micro-channels (to form large numbers of nano-liter scale reaction chambers), is being developed in PDMS. LoC allows the detection and isolation of cells, nucleic acids, and proteins; provides faster detection of pathogens and biohazards; and accelerated clinical diagnostics. Earlier, such systems were made in glass or silicon that was expensive and had problems of clogging of the channels and cross-contamination, whereas a PDMS based system is cost-effective and disposable.

This paper describes the salient features of soft-lithography, the fabrication methodology, PDMS properties and surface modification, along with its applications in various disciplines of science and technology. The R&D carried out in the author's laboratory will also be discussed.

2. Poly-dimethylsiloxane and its properties

Polydimethylsiloxane, an inexpensive elastomer, is indeed one of the most attractive materials in microchip technology. It consists of two components; a base and a

curing agent. Their mixture is cured by an organometallic cross-linking reaction. The siloxane base oligomers contain vinyl groups, whereas the curing agent contains a platinum-based catalyst that catalyses by addition of a SiH bond across the vinyl groups, forming Si-CH_2-CH_2-Si linkages. The mechanical properties of PDMS can be controlled by manipulating the composition of the uncured material and the curing conditions. For example, increasing the ratio of the curing agent to the base enhances cross-linking in the polymer and results in a harder and rigid elastomer, whereas curing at elevated temperatures accelerates the cross-linking reaction.

PDMS is flexible and optically transparent down to 230 nm, which makes it suitable for detection over a wide range of wavelengths (and therefore compatible with many optical detection methods). It is suitable for biological studies because it (i) is impermeable to water but is permeable to gases, a property that is useful for supplying oxygen to cell cultures in closed systems and (ii) is non-toxic to cells and does not damage living cells, qualifying it as a good biocompatible material for cell patterning on various substrates. PDMS provides a surface that has a low interfacial free energy (21.6 dyne/cm^2) and good chemical stability; most molecules or polymers do not adhere irreversibly to, or react with, the surface of PDMS. The PDMS elastomer has good thermal stability (up to ~185°C in air) and consequently, it can be cured thermally. PDMS can readily seal to itself and to other flat surfaces such as glass, silicon, polystyrene, PMMA and other polymers, reversibly by conformal contact (*via* van der Waals forces), or irreversibly if both surfaces are oxidized by a plasma for a short duration before bringing them into intimate contact with each other under ambient conditions [3]. The major advantage of PDMS is the ease with which micro-systems can be fabricated and assembled (e.g., bonding) compared to conventional glass and silicon [4] systems that require a clean room environment, expensive photolithographic and not too friendly anodic or thermal bonding techniques.

In spite of the many advantages of PDMS, its application in microfluidics or in micro-contact printing has not been free from problems. The CH_3 groups at the tail in the repeating -OSi$(CH_3)_2$- units of PDMS make its surface inherently highly hydrophobic [5], with a water contact angle 100–115º. Thus, microchannels in PDMS are difficult to fill with aqueous buffers. In addition, PDMS absorbs a number of nonpolar organic solvents such as toluene and hexane and hydrophobic analytes, causing fouling and swelling of the material [1,4]. Furthermore, it makes the surface prone to the non-specific adsorption of proteins and cells [4].

The cured PDMS has a very low surface energy and is inherently inert in character, making it difficult for chemical species to interact with its surface. Changing the surface characteristics of PDMS by an oxidation process results in a higher surface energy, and the adhesion characteristics of the material are enhanced markedly [6].

3. Surface characteristics of PDMS

Several oxidation techniques [6], such as oxygen plasma, corona discharge, Ar^+ plasma, UV light, etc. have been used to alter the surface properties of PDMS from a hydrophobic (contact angle >100º) to a hydrophilic (contact angle <5º as shown in Fig. 1) wherein methyl (Si-CH_3) groups are replaced with hydroxyl (Si-OH) groups. But the hydrophilicity of oxidized PDMS is short-lived and it quickly regains its native hydrophobic character [6, 7]. However, the rate of recovery depends strongly on the ambient and storage conditions [6, 8]. It can be seen from Fig. 2 that the auto-phobic recovery of plasma oxidised samples stored under water and in an inert atmosphere is much slower than those samples kept in an ambient surrounded by air.

The recovery process has been attributed to the rearrangement of surface molecules occurring due to the migration of low molecular weight species of hydrophobic nature to the upper surface from the bulk, lowering the surface free energy and bringing it back to thermo-dynamical stability [9]. The contact angle and SEM are the most widely used techniques to evaluate PDMS surface properties.

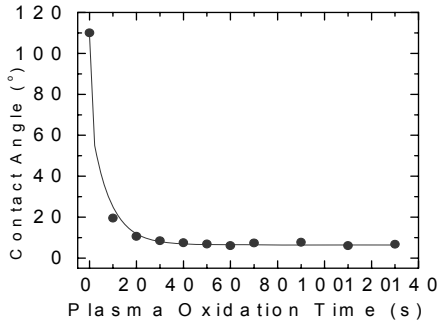

Fig. 1. Contact angle vs. plasma oxidation time, showing the transformation of a highly hydrophobic PDMS surface into a completely hydrophilic surface.

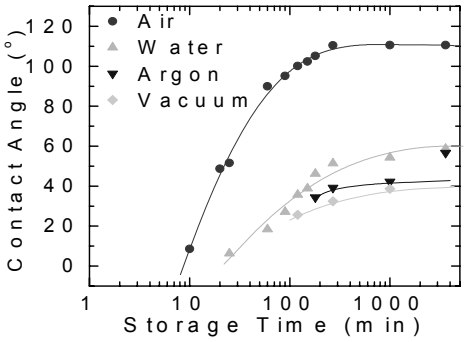

Fig. 2. Contact angle as a function of storage time for PDMS samples kept in air, water, and vacuum and in an inert atmosphere of argon after plasma oxidation (rf power = 50 W and oxygen pressure=0.1T).

4. Elements of soft-lithography

4.1 Creation of masters

The replica of the structures to be patterned in PDMS are first created on silicon substrates using standard photolithography and etching techniques, as used commonly in MEMS technology. It is required, generally, to create feature dimensions smaller than 5 microns. However, larger dimensions (varying from a few tens to hundreds of microns) can be made more conveniently using SU-8 photo-resist. For the fabrication of masters having feature sizes greater than 20 μm, the mask can even be generated on overhead transparencies using high-resolution laser printing technology available commercially. This procedure is rapid and less expensive, which greatly reduces the requirement for expensive optical lithography and stringent clean room conditions. However, chrome masks used in photolithography are required for features less than 5 μm in size.

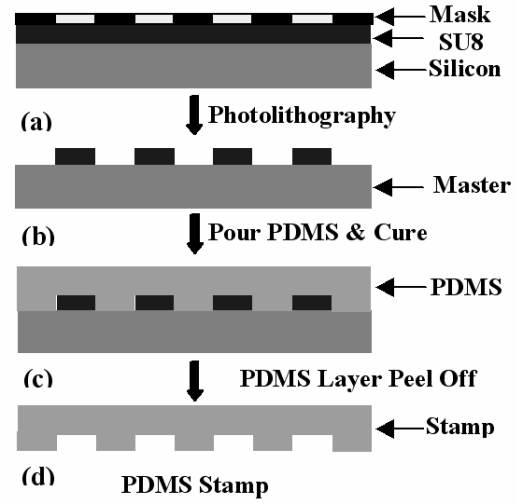

Fig. 3. Process flow for making a PDMS stamp from a silicon master or a master made using SU8.

In the soft-lithography process, photolithography is used only once for the fabrication of the masters. Generally, optical lithography, micro machining, e-beam writing, etc. can be used to make masters. In addition, other commonly available relief structures such as diffraction gratings, TEM grids, polymer beads assembled on solid supports, and relief structures etched in metals can also be used as masters. A schematic of the process flow used to make a PDMS stamp from a master is shown in Fig. 3. In place of the master made in SU-8 (Fig. 3b), a master in silicon made using MEMS technology can also be used.

4.2 Elastomeric stamp

The elastomeric stamp, which is the key element in soft-lithography, is prepared by casting the liquid pre-polymer, i.e. PDMS elastomer, against a master that has a patterned relief structure. It is desirable to treat the master with fluorinated silanes, to prevent its irreversible bonding with the PDMS prior to the elastomer moulding. The PDMS prepolymer is mixed together in the ratio of 10:1 v/v of base and curing agent, and subsequently degassed in a vacuum dessicator at ambient temperature for several hours, to remove the air bubbles from the PDMS solution. The homogenous mixture is poured into the master and cured at 60-70 °C for several hours. The liquid PDMS pre-polymer subsequently polymerises and conforms to the pattern of the master, replicating the features with high fidelity (Fig. 3c). The cured PDMS (now called a *stamp*) is peeled-off from the master (Fig. 3d). The low surface free energy and elasticity of the PDMS allow it to be released from the master without damaging the master or itself. The same master can be used repeatedly to produce stamps and moulds. The stamp exhibits high fidelity and dimensional stability (±2%) and, being elastomeric in nature, it can contact non-planar surfaces conformally. Consequently, lithography can be done on curved surfaces. This is the distinct advantage of soft-lithography with an elastomeric stamp over conventional lithography. The same PDMS stamp can be used to prepare many impressions over a period of several months, without noticeable degradation in its quality and performance.

However, there are some problems associated with PDMS stamps that are important and should be taken care off. The softness of PDMS puts a limit on the aspect ratio (height to length ratio) of the relief features and must be between 0.2 and 2 in order to obtain defect-free stamps or moulds [1]. When the ratio is too high or too low, the stamp may deform and generate defects in the pattern. The most common problems are sagging of stamps and pairing of stamp features, as shown in Fig. 4.

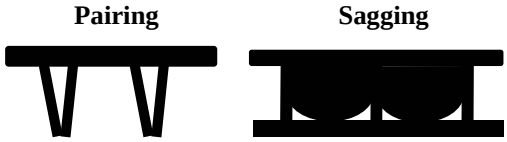

Fig. 4. Pairing and sagging of a PDMS stamp.

The former is due to compressive forces between the stamp and the substrate. This limits the use of μCP for patterns with widely separated features, unless the stamp is mounted on a rigid support or non-functional posts are introduced in the design [1]. The latter is observed when the raised features are long and not separated widely. The paired lines could be restored by washing the surface with a <1% aqueous solution sodium dodecylsulfate followed by rinsing in heptane [10].

4.3 Ink

A number of chemicals can be used as 'ink' solutions that adsorb irreversibly to the elastomeric stamp and can subsequently be transferred onto specified regions of the substrate. A range of self-assembly monolayers (self assembly is the spontaneous organization of sub-units such

as molecules into a stable structure at or close to thermodynamic equilibrium) forming solutions such as organic and inorganic compounds, colloids, sol gels, proteins and cells can be used as ink. Alkane thiols ($X(CH_2)_nSH$) are the most widely used compounds to fabricate patterned structures on various metal films like gold, silver, copper, palladium etc. The thickness of a SAM depends on the number (*n*) of methyl groups in the alkyl chain, whereas the surface properties depend on the head group, X that can be easily modified by a chemical route. The terminal group X can be fluorocarbons, acids, amines, alcohol etc. Thiols form highly ordered SAMs by the spontaneous bonding of sulphur atoms onto the metal surfaces where the alkyl chains are trans-extended due to van der Waals interactions and are, generally, tilted at some angle from the normal to the surface. SAMs exhibit many attractive characteristics: ease of preparation, good stability under ambient conditions, relatively low densities of defects in the final structures and amenability to application in controlling interfacial (physical, chemical, electrochemical and biochemical) properties.

Long-chain alkanethiols $\{-S(CH_2)_nCH_3\}$, such as hexadecanethiol (HDT, n=15), octadecanethiol (ODT, n=17), or eicosanethiol (ECT, n=19) are commonly used as inks on noble metal surfaces [11]. Their SAMs are highly protective etch resists on noble metals against common etchants, and also promote hydrophobic adsorption of most proteins. On the other hand, SAMs of ethyl glycol terminated thiols resist the adsorption of proteins.

Surfaces with SAMs of alkanethiolates on Au films are well understood and are, indeed, widely used as model systems to study interfacial phenomena such as wetting [12], nucleation, protein adsorption [4,13] and cell attachment. The closed packed trans-extended alkyl chains on (111) Au are tilted at ~30^o from the normal to the surface. Although the SAMs formed on silver are more ordered than on gold, cytotoxicity of the Ag^+ released on exposure to air and other oxidants limits their use in biological experiments involving living cells. The alkyl chains on Ag are oriented nearly perpendicular to the film surface.

4.4 Sealing

Early microfluidic systems have been fabricated in silicon/glass, where micro-channel networks were defined in the substrates using photolithography and micromachining. The channels were closed by sealing the substrates having relief features against a flat surface (such as glass or silicon) using anodic or fusion bonding (that requires applying a combination of high pressures, temperatures, and voltages for an extended period). Silicon based systems were adopted because they relied on the technology available in the microelectronics industry. Such devices were not only expensive but were not very suitable for rapid prototyping and quick exploratory work. As these devices were irreversibly sealed, they could not be dismantled and cleaned in case of clogging or to eliminate the possibility of cross-contamination.

As pointed out earlier, PDMS can readily seal either reversibly or irreversibly to itself or to a variety of substrates such as glass, silicon nitride, silicon, polystyrene, polyethylene, etc., without distortion in the relief features. Such attributes make PDMS ideally suited for fabricating topologically complex miniaturised systems.

The reversible sealing is realized by conformal contact between the PDMS replica and a flat piece of PDMS or glass, which is provided by simple van der Waals contact. Although such seals are watertight, they cannot withstand pressures greater than 5 psi, whereas irreversible sealing is realized by exposing the PDMS replica (for example, a network of channels) and a flat piece of PDMS or any other materials mentioned above to air plasma for a short duration, followed by quick conformal contact of the two oxidized surfaces. The condensation of the silanol groups on the oxidized surfaces results in the formation of an irreversible seal that can stand up to 50 psi pressure.

In the following section, the work carried out at National Physical Laboratory will be discussed.

5. Micro contact printing on metal films

5.1 Metal surface patterning using μCP

In the present work, the self-assembled monolayer of hexadecanethiol (HDT) was selectively transferred to the specific regions of the substrate with a PDMS stamp, which was prepared by casting the prepolymer of PDMS against a master mask that had a patterned relief structure on its surface. The masters used to cast stamps were prepared either by conventional photo-lithography (using AZ Shipley positive photo-resist or SU8 photo-resists) or by etching patterns in silicon substrates. The prepolymer, on thermal curing at 60 °C for 4 hours, could be easily peeled off from the master. The quality of the relief structure on the stamp was examined under a scanning electron microscope (SEM) and was found to have a very high fidelity (±2% of the master).

The elastomeric stamp was ‘inked’ with an ethanolic 0.01 mM solution of HDT and was dried in a dry nitrogen stream. The ‘ink’ molecules were transferred to the surface of the metal films by physical contact of the stamp for a short duration (~30s). The gold/silver films (100-1000 Å thick) were deposited by thermal evaporation in a vacuum coating unit on glass or silicon substrates, and were primed with a thin titanium coating (50-100 Å).

The schematic of patterning of metal surfaces using micro contact printing is depicted in Fig. 5.

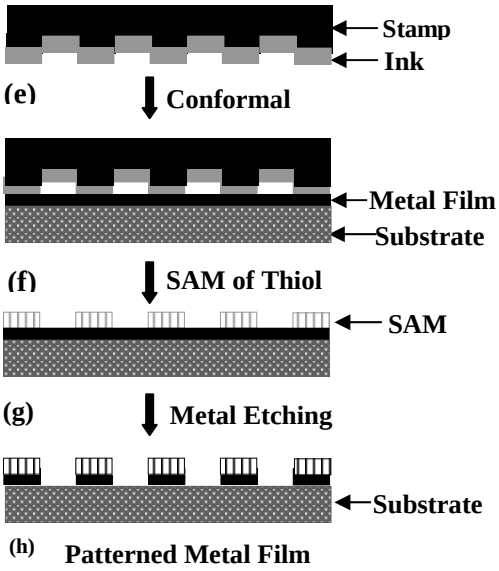

Fig. 5. Process flow for patterning of a metal film using Micro Contact Printing.

The hexadecanethiol molecules chemisorb on the metal surface with the loss of hydrogen and the sulfur atoms get bonded to the metal surface. This leads to the formation of a well-organized SAM structure that changes the wetting properties of the substrate and acts as a 'nanoresist' against wet etchants used for etching metal films. The un- derivatized regions, i.e., "non-inked" regions on the metal films can be easily etched by wet chemical etchants.

An aqueous $Na_2S_2O_3$, $K_3[Fe(CN_6)]$ and $K_4[Fe(CN_6)]$ solution was used to etch silver and gold films, whereas $FeCl_3$ (pH=7.4) and diluted HF (<1%) were used to etch palladium and titanium respectively.

A typical SEM picture of a pattern created in a silver film on glass, with sharp edge definitions (feature sizes <3μm), is shown in Fig. 6.

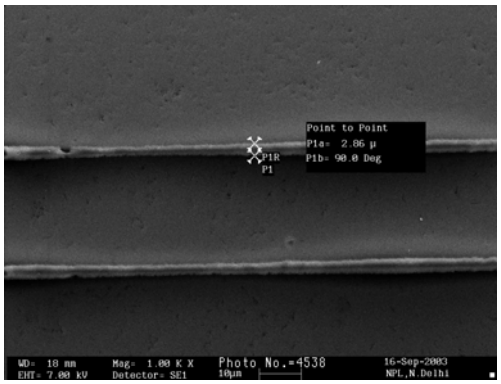

Fig 6. Etched patterns in a silver film on a glass substrate with a feature size < 3 μm, using a PDMS stamp.

An optical photograph of an etched pattern with features ranging from a few tens microns (<25 μm) to several hundred microns (>100 μm) over a large area (a few cm^2), created on a silver coated silicon substrate, is shown in Fig. 7.

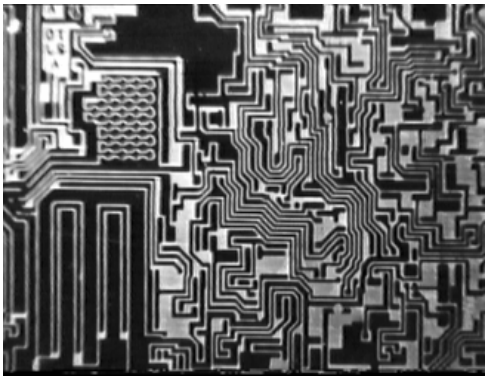

Fig. 7. Etched Patterns in a silver film on a silicon substrate with feature sizes from 25 – 100 μm, using μCP.

5.2 Micro-patterned surfaces with contrasting physical and chemical properties

Micro-patterned surfaces with contrasting wetting and de-wetting properties could be made by n-alkane thiols with different functional groups by sequential deposition on different substrates (metals films, silicon, quartz, glass etc.) using μCP. The terminal group of the alkane thiolates dominates the surface properties. For example, the thiolated regions with methyl terminated groups exhibit a highly hydrophobic character whereas regions with hydroxyl terminated groups exhibit a strongly hydrophilic character.

The co-patterned SAMs are highly stable in ambient conditions, and have good control over the interfacial properties. Such systems provide excellent contrasting wetting properties (Fig. 8) and can be used for selective nucleation and growth studies related with wetting, adhesion, corrosion, lubrication, protein adsorption etc.

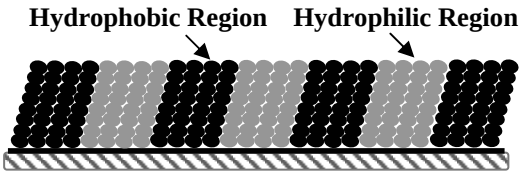

Fig. 8. Schematic of alternate hydrophobic and hydrophilic regions on gold film.

SAMs with different functional groups at the termini (fluorocarbons, acids, amines, alcohol, glycol etc.) of the alkyl chains of alkane thiols are co-patterned to create pattern structures with contrasting wetting and de-wetting properties. First, a monoalyer of alkane thiol with a methyl terminal group (e.g., HDT) was transferred to the selective regions of a gold/silver-film deposited on a substrate by μCP, as described in an earlier section. Subsequently, the

substrate was immersed in an ethanolic solution of mercapto-hexadecanoic acid (MHA) that formed the SAM in the un-derivatized metal-coated regions. Poly ethylene glycol (PEG), which is superior in terms of the wetting property (hydrophilic), was also used as an alternative to MHA. The resulted structure showed highly hydrophobic and hydrophilic regions derivatized with a methyl and an acid group respectively, as shown in Fig. 9.

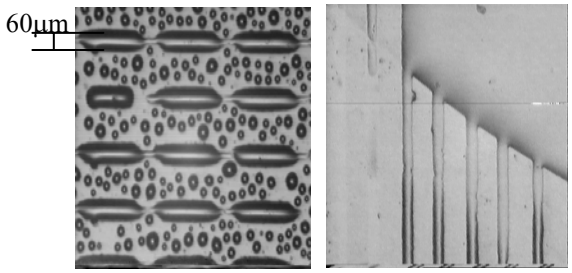

Fig. 9. Micrograph of contrasting wetting on a gold film using MHA *(left) and* PEG *(right).*

The former form water droplets while the latter is wetted by water. Similar patterned surfaces have also been made on hydroxylated glass/silicon surfaces, by sequential deposition of SAM of alkyl trichloro-silane and PEI respectively. Such patterned structures can be used for the selective nucleation and growth of inorganic crystals; optical wave-guides and micro-lenses; selective adsorption of proteins and cells [4,13] etc.

Therefore, micro-patterned surfaces (using μCP) have applications in microelectronics and the life sciences. For example, multi-layers of TiPdAg are standard Ohmic contacts for silicon based devices, and patterning could be done by μCP rapidly and with ease. It can be directly used for making contacts on solar cell and photodiodes, whereas metal films with an array of alternating hydrophilic and hydrophobic lines or islands can be used to immobilize proteins and for cell culture.

6. Microfluidic channel network in PDMS for handling nano-litre solutions

Masters having microfluidic channel networks were made in SU-8 photo-resist using a standard photolithography technique. To produce the replica, a pre-polymer of PDMS was poured into the master, and cured at 60 °C for 12 hours. The PDMS stamp replicated the features of the master with high fidelity. The elasticity and low surface energy of PDMS allowed it to be released from the master without damaging the master or itself. Flat PDMS blocks were made by molding against a flat surface.

A closed microfluidic network was constructed by sealing the two blocks, one having the relief structure and the other a flat surface. For irreversible sealing, the samples were placed on the water-cooled cathode in an oxygen plasma chamber and were subjected to an RF field of 70 W for 30 seconds with flowing oxygen at 50 sccm. The two samples with activated surfaces facing each other were brought into contact with each other immediately.

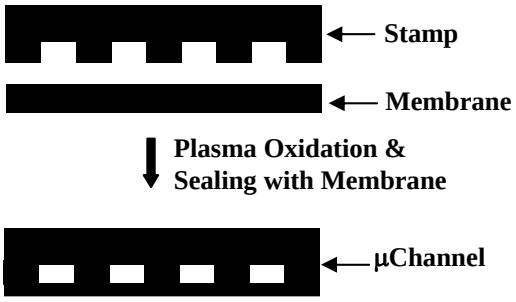

Fig. 10. Schematic of the irreversible sealing process used to construct a closed microfluidic channel.

At this point, no external force was applied to the samples to assist the bonding procedure. For reversible sealing, the two virgin surfaces are brought in conformal contact with each other by an external force.

Two structures, one a Y-shaped microfluidic and the other consisting of 10 microfluidic channels with independent access ports were made. The layout of the Y-shaped microfluidic device is shown in Fig. 11. The main channel is 360 μm in width and 1.2 cm in length. The two input channels are ~180 μm in width. All the channels are ~50 μm deep. The cover plate, which was in alignment with the channels, has two holes. These act as macroscopic reservoirs to hold the fluids. It can be clearly seen that the flow inside the channels is well confined and the diffusion takes place only at the interface.

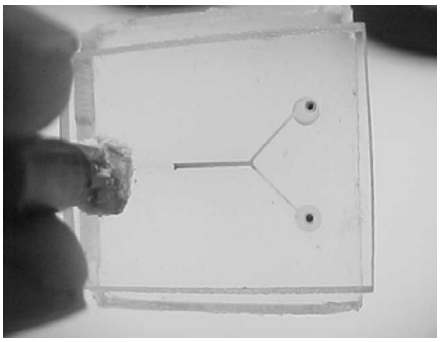

Fig. 11. Optical photograph of a Y-shaped micro - fluidic channel with access ports.

Fig. 12 shows the fluid flow in a micro channel network (channels: 40-80 μm wide, 75 μm deep and 1.4 cm long) with independent access ports. It can be seen that different channels can be filled with different fluids without any inter-mixing. The fluid flow was recorded with an optical microscope using a CCD camera. The devices were capable of handling nano-litre solutions with ease and rapidity.

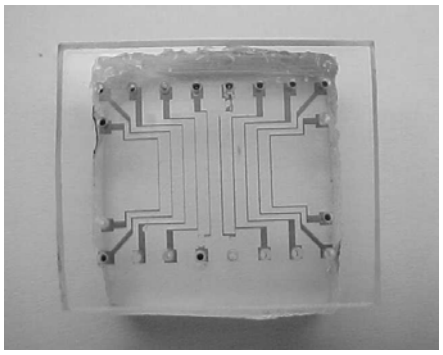

Fig. 12. Optical Photograph of a system having ten independent microfluidic channels with separate access ports.

The structures shown above are indeed basic building blocks for microfluidic systems and devices. For example, the channel network can be used to construct functional components and for the patterning and selective deposition of bio-molecules. The fluids containing the species (e.g., bio-molecule) to be patterned would flow through the channels and create patterns in the bound surfaces through covalent attachment, adsorption or the dissolution of the material already present inside the channels. The non-communicating multi-channel network with independent access ports would allow for simultaneous patterning of different proteins in well-defined regions adjacent to each other [14].

7. Conclusions

Micro contact printing has been used to create patterns on Au, Ag, Ti and Pd surfaces, with feature sizes ranging from sub-μm to tens of μm, that has potential applications in solar cells and photodiodes. Surfaces with contrasting wetting and de-wetting properties have been realized by sequential deposition of methyl and hydroxyl terminated allkanethiols that may be used to immobilize proteins and cells selectively. Microfluidic channels in PDMS were fabricated and sealed against a flat PDMS block by plasma oxidation to construct a micro-channel (μC) network. The surface properties of μCs have been effectively controlled by rendering highly a hydrophobic PDMS surface into a hydrophilic surface for laminar fluid flow. Such systems are being examined for the direct transfer and patterning of biomolecules (proteins and cells) and nano-particles to the specific regions bound by channel walls or through raised relief structures on an elastomeric stamp. The PDMS based micro-systems have applications in immunoassays and microchip cell-based bioassays.

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