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A reusable high aspect ratio parylene-C shadow mask technology for diverse micropatterning applications

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Abstract

In this paper, we present a low cost, flexible and reusable parylene-C shadow mask technology for diverse micropatterning applications. The smallest feature size of 4 μm is demonstrated and the technology is scalable up to full wafer scale. With the addition of SU-8 pillars, we also demonstrate multimask processing with an alignment accuracy of about 4–9 μm. To achieve features with fine resolution, a low temperature and high aspect ratio (>8:1) parylene etch process is also developed. Utilizing this shadow mask, we successfully patterned proteins and cells on various surfaces (glass, PDMS, methacrylate). High pattern flexibility (structures with different shapes and dimensions are successfully patterned) and patterning on curved PDMS surfaces are also demonstrated. This technology has potential applications for patterning proteins, cells and organic transistors on conventional and/or unconventional substrates.

Keywords: Parylene-C; Flexible shadow masks; High aspect ratio polymer etching

1. Introduction

Classical microfabrication based on optical lithography has limitations for applications such as patterning organic materials (solvent incompatibility), patterning on fragile (released) MEMS devices, patterning of non-traditional materials (proteins and cells), and patterning on plastic substrates (that cannot withstand high temperatures) and on non-planar surfaces. Hence, shadow mask technology is gaining impetus as an alternative micropatterning technique for diverse applications on conventional and unconventional surfaces.

Shadow masks can be classified as active or passive shadow masks. The difference between them is that the aperture size of the active shadow masks are adjustable within demand [1,2] whereas the passive shadow masks, which include most current shadow masks, have a fixed aperture size. Previously reported passive microstencils, which are made of rigid or polymeric membranes, have various limitations. For instance, Si, Si_3N_4, TEM grid and stainless steel shadow masks are rigid and brittle, require complicated and expensive processing steps [3,4]. In addition, they lack the precise pattern definition and the pattern flexibility to different pattern dimensions due to the gap between the stencil and the substrate [5–8]. Elastomeric microstencils (such as PDMS), on the other hand, are not easy to handle, and have difficulty in achieving mechanical alignment and lack high resolution [9]. Shadow masks made of SU-8 polymers [10] are also not suitable for wafer scale patterning applications since their high residual stress makes them buckle. Microstencils made of JSR THB-430N negative UV photoresist will result in enlargement of features due to their non-straight sidewall profiles [11]. Furthermore, dry lift-off method as demonstrated by Ilic and Craighead is limited to thin films of single use and to surfaces compatible with microfabrication technologies (silicon and glass) [12].

In this paper, we present a flexible, reusable, transparent and biocompatible parylene-C microstencil technology as illus-

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Fig. 1. The parylene shadow mask being peeled off a wafer after fabrication.

trated in Fig. 1. To realize this stencil, a novel low temperature parylene-C etch process is developed to create the high aspect ratio (HAR) structures with the use of an inductively coupled plasma (ICP) tool. The potential applications of this stencil technology are numerous including patterning for organic electronics, patterning of proteins and cells and patterning on topographically rough, curved and unconventional surfaces and in fabricating metamaterials.

2. Parylene-C deposition

Parylene, poly-*para*-xylylene, is widely utilized in the medical and electronics industries as a conformal pinhole free coating. Due to its high mechanical strength (tensile strength of 3.2 GPa) it is being increasingly utilized as a structural layer as well as a flexible substrate [13,14]. Up to a certain thickness (20 μm) the parylene films are flexible and will conform to curved surfaces and also have the high mechanical strength and robustness compared to PDMS stencils making them reusable [21]. The flexible shadow mask is fabricated from parylene-C which is deposited at room temperature and we first detail its deposition process in the following section.

Polymerizations of polymer materials are typically done in solution form or gas/vapor phase form with/without the assistance of plasma [15]. Parylene deposition is a chemical vapor deposition (CVD) process, which is done at 25 mTorr and at room temperature (25 °C). Parylene deposition process has three main stages. The first stage is vaporization process, where a solid parylene dimer is vaporized at a temperature of 175 °C. The second stage is the pyrolysis process, during which vaporized parylene gas moves slowly into the pyrolysis chamber, and the parylene gas is decomposed into the parylene monomer at a temperature of 690 °C. The last stage is the deposition process where the parylene monomers move slowly into the deposition chamber and get adsorbed on the substrate surface and polymerize. The steps of parylene deposition are illustrated in Fig. 2.

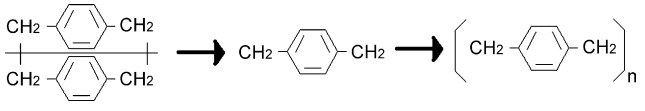

Fig. 2. The parylene deposition sequence.

During the polymerization process, the monomer in the deposition chamber is first adsorbed on the substrate, then surface migration and bulk diffusion of monomers take place, finally the chemical reaction between the monomers form the film. The mean free path of parylene monomer in the deposition chamber is in the order of 0.1 cm during this process which results in conformal deposition. Since the polymerization process occurs at the room temperature, the deposited parylene films are relatively stress free. Parylene shadow mask requires a fairly thick membrane, ~10–20 μm so that it is reusable for micropatterning applications.

3. Fabrication of the parylene-C shadow mask

To fabricate the flexible microstencil, first, a 10–20 μm thick parylene is deposited on a silicon wafer (PDS2010, Specialty Coating Systems, Indianapolis, IN). Prior to the deposition of aluminum, we routinely roughen the parylene surface to enhance adhesion of aluminum onto the parylene surface utilizing the inductively coupled plasma reactor (Plasmatherm 790) under the following conditions (RF bias power = 100 W, source power = 150 W, O_2 flow = 50 sccm (standard cubic centimeters per minute), Ar flow = 20 sccm, time = 30 s, pressure = 20 mTorr, temperature = 25 °C). Then, a 2000 Å thick aluminum hard mask is deposited using sputter deposition. The patterns are next generated by conventional photolithography method using a positive photoresist (Microposit S1813, Shipley Company) and aluminum is etched (using photoresist as a mask) in aluminum etchant type A (Transene Company Inc., Danvers, MA) at 50 °C for 30 s. Next utilizing the Al as a hard mask, we etch through the parylene layer in an ICP etcher (Plasmatherm 790). After the ICP etch, the Al hard mask is removed in aluminum etchant type A at 50 °C for 2 min. The parylene shadow mask is next peeled off the wafer as shown in Fig. 1 and is ready to use. As a side note, prior to parylene deposition, we routinely use HMDS as an adhesion promoter since conventional adhesion promoters for parylene-C such as A-174 silane tend to create strong adhesion between the film and the substrate and hence cause the film to tear upon peeling. We have demonstrated that both 10 μm and 20 μm thick parylene-C films are flexible and reusable. For small area applications (i.e. 10 mm × 10 mm) we recommend using 10 μm thick stencils whereas for large area applications (three inch wafer level), the 20 μm thick film is recommended even though it is slightly less flexible.

For patterns with large dimensions (in excess of 200–300 μm) that do not require fine (2–3 μm) resolution, we fabricate the shadow mask with a room temperature ICP etch since lateral etching is not a major concern. Furthermore, while fabricating stencils with fine features (<10 μm), one requires an anisotropic etch and hence, we have developed a novel high aspect ratio parylene etch process which is detailed in the next section.

4. High aspect ratio etching of parylene-C

Parylene is gaining popularity as a unique low temperature material for many biomedical and non-biomedical applications [16,17]. One of the current needs for the parylene micromachin-

Table 1
ICP etch recipes for low temperature (5 °C) and low pressure (5 mTorr) parylene-C etching

Etch #	Etch rate (μm/min)	RF bias power (W)	Source power (W)	O_2 (sscm)	Ar (sccm)
a	1.7	250	400	20	0
b	1.0	100	400	10	10
c	0.5	100	150	10	10

ing community is a high aspect ratio etching process. Meng et al. [18] utilizing a DRIE tool, obtained aspect ratios of up to 3:1, moreover to create reusable stencils with fine features, stencils with higher aspect ratio structures are required. It is possible to reduce the isotropy of a reactive ion etch process by reducing the etch temperature which is commonly done by etching silicon at low temperatures (∼−100 °C). Moreover, for etching polymers such as parylene-C, reducing the etch temperature down to 5 °C serves a similar purpose. Using an ICP reactor (Plasmatherm 790), we developed multiple recipes (Table 1) with fast etch rates and anisotropic profiles (>8:1). The parylene film shown in Fig. 3(a) with a thickness of 55 μm is etched with the recipe "b" in Table 1 and the one shown in Fig. 3(b) with a thickness of 10 μm is etched with recipe "c" in Table 1 and they both display almost vertical sidewalls. We were able to etch a 55 μm thick parylene film through an opening of 6 μm which is equivalent to an aspect ratio of about 9:1. Aluminum was used as the hard mask during the ICP etching process, which worked well except the fact that it sputtered during the etching process and created residues as seen in Fig. 3(b). We are currently investigating means to address this issue.

5. Surface properties of parylene-C

As-deposited parylene-C, similar to PDMS, displays hydrophobic properties with a contact angle of ∼98 °C. The as-deposited hydrophobic parylene surface seals extremely well to other hydrophobic surfaces. Furthermore, it does not adhere well to hydrophilic surfaces, a property that is important while using the parylene-C as a shadow mask. Accordingly, we have characterized the contact angle and the stability of parylene sheets in aqueous environments over time. We have submersed an as-deposited parylene sheet into deionized water for 3 days (which may be the case for multiple patterning and rinsing experiments) during which the surface maintained its hydrophobic behavior as seen in Fig. 4(a). For various applications (such as patterning proteins and cells), one would prefer to have a hydrophilic surface and require that the surface maintains such property

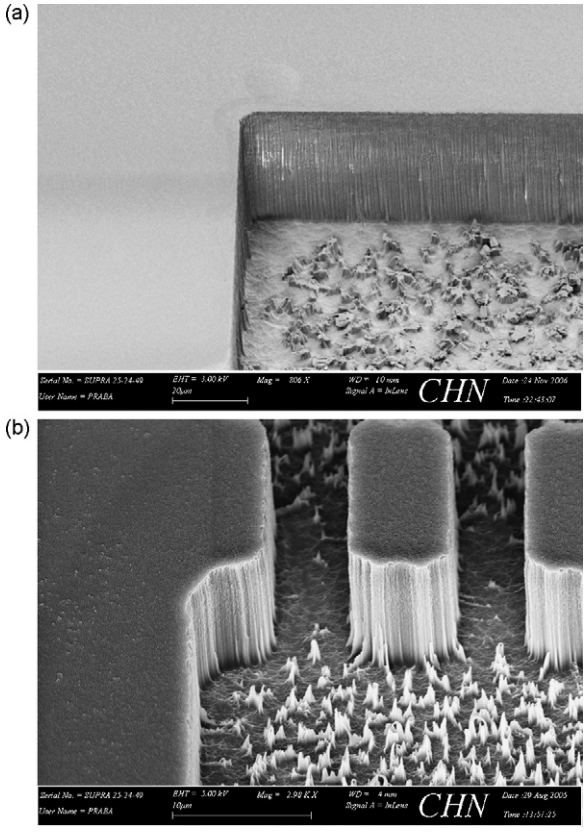

Fig. 3. Anisotropic etch profiles from (a) 55 μm thick parylene and (b) 10 μm thick parylene.

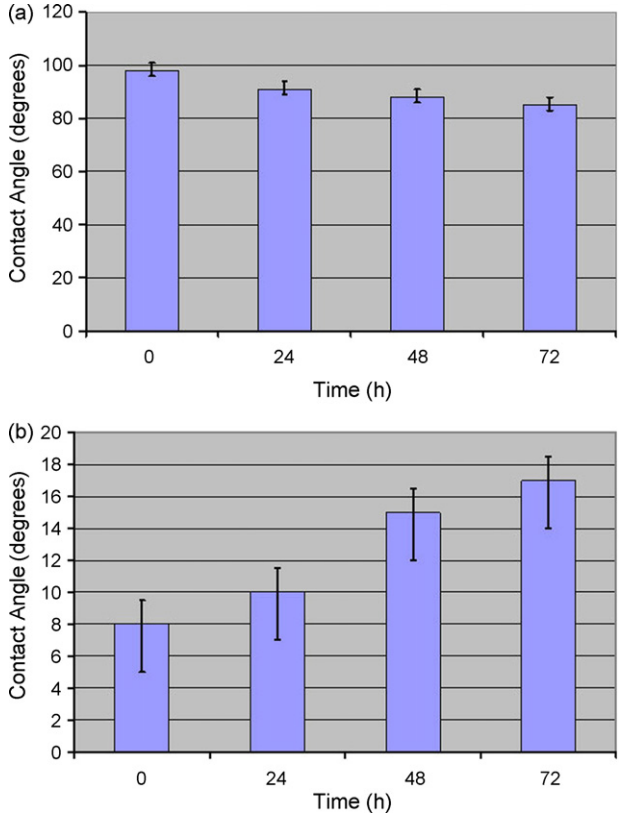

Fig. 4. (a) Contact angle measurement of as-deposited parylene-C surface in aqueous environments up to 3 days. (b) Contact angle measurement of hydrophilic parylene surface in aqueous environments up to 3 days.

after being exposed to aqueous environments. Utilizing a short O_2 plasma treatment (pre-metal surface roughening recipe from Section 3), the as-deposited hydrophobic parylene surface can be converted into a hydrophilic surface. Similar to the hydrophobic stability test, hydrophilic parylene surface was also tested in deionized water over 72 h. Unlike PDMS which becomes hydrophobic within a few hours after plasma treatment, parylene surface remained hydrophilic days after plasma treatment as displayed in Fig. 4(b). A side note is that the roughening recipe utilized to improve the adhesion of aluminum to the parylene surface renders the top parylene surface hydrophilic. Moreover, the bottom side of the stencil remains hydrophobic during processing and can seal easily to many hydrophobic surfaces in a conformal manner.

6. Micropatterning applications of the parylene shadow mask

Fig. 5 illustrates the sequence of steps for micropatterning utilizing the parylene shadow mask. In Fig. 5(a) we illustrate the parylene shadow mask after being peeled off a wafer. First, we place the microstencil on a substrate where the patterning is needed as shown in Fig. 5(b). Then, we deposit the material (e.g. deposit 1500 Å gold or 1500 Å aluminum) over the shadow mask as seen in Fig. 5(c). Next, we peel off the shadow mask from the substrate which results in patterned microstructures on the substrate as seen in Fig. 5(d). The peeling process does not cause damage to the desired dimensions of the micropatterns in terms of shape, size and aspect ratio. Finally as illustrated in Fig. 5(e), the parylene shadow mask is ready for reuse after removing the deposited material. For instance, to remove 1500 Å of aluminum, we place the stencil into aluminum etchant (aluminum etchant type A) for 2 min at 50 °C. Parylene stencil can be utilized for patterning many low temperature deposited materials, and we have demonstrated a few examples in patterning proteins and cells (utilized a 10 μm thick parylene stencil), metal layers (utilized a 10 μm and 20 μm thick parylene stencil), patterning on curved surfaces (utilized a 10 μm thick parylene stencil) and multistep patterning (utilized a 20 μm thick parylene stencil), and the results are described below.

6.1. Micropatterning of proteins and cells

Patterning of proteins and cells has previously been demonstrated with a parylene-C film by Takeuchi [9] and Craighead [12] on traditional surfaces. Since both groups utilized a very thin parylene layer (between 1 μm and 2 μm), their approach was limited to single use where the parylene membrane tore apart upon peeling and also the approach was limited to traditional surfaces onto which parylene can be deposited and etched from (silicon and glass). In our approach, we remove the parylene-C shadow mask from the surface where it is fabricated on and then apply it to any desired surface.

Parylene-C is a well-known biocompatible material utilized for encapsulating implantable devices. Using our parylene stencil, we successfully patterned proteins on polystyrene and methacrylated glass surfaces as seen in Fig. 6. Since parylene is a relatively inert material, one can wash away the protein solutions and can reuse the parylene stencil multiple times. As

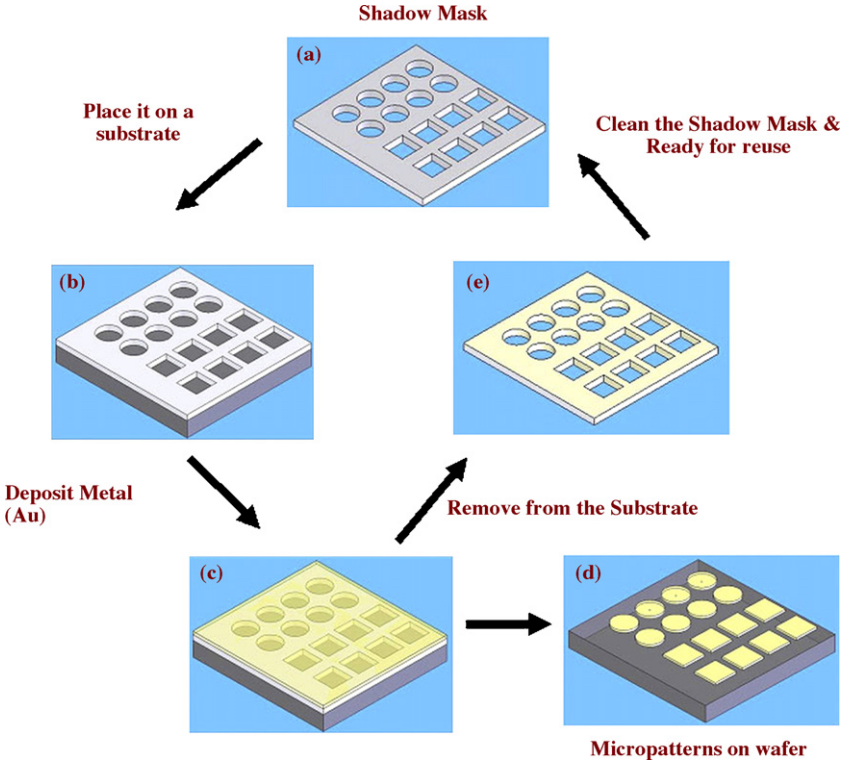

Fig. 5. Sequence of steps for micropatterning using reusable parylene shadow mask: (a) shadow mask as fabricated, (b) place it on a substrate, (c) deposit metal (Au), remove the shadow mask from the substrate (d) and etch the deposited metal (e) and repeat steps (a-d).

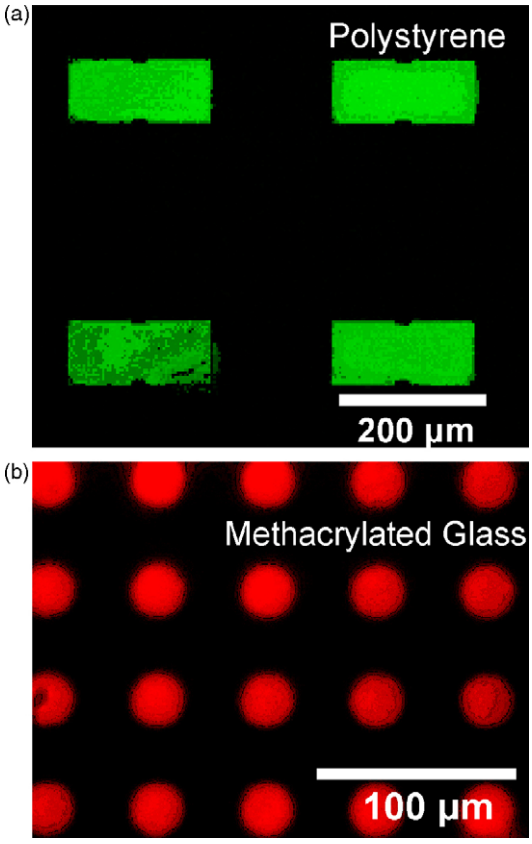

Fig. 6. Fluorescent images of proteins patterned on (a) polystyrene and (b) methacrylated glass surfaces.

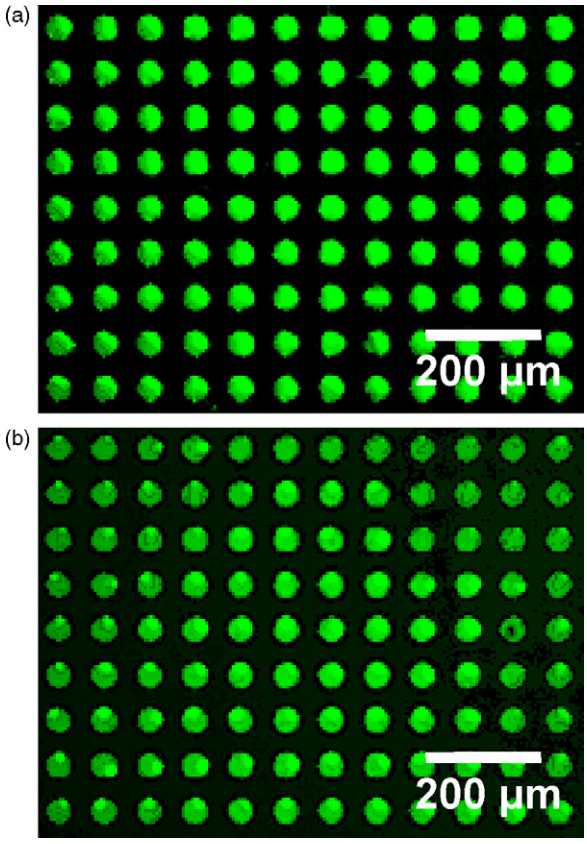

Fig. 7. FITC-BSA was patterned on PDMS after (a) 1st patterning and (b) 9th patterning.

shown in Fig. 7(a), we were able to pattern FITC-labeled BSA protein on a PDMS substrate using the stencil. The patterning was repeated nine times, during which the resolution was maintained as seen in Fig. 7(b). Following protein patterning, we next demonstrated applications of the parylene stencil in tissue engineering and have successfully patterned NIH-3T3 fibroblasts (as seen in Fig. 8) and other cells types including AML12 hepatocytes and mouse embryonic stem cells on PDMS surfaces. Next, we fabricated a cylindrical PDMS slab and utilizing our flexible parylene stencil, patterned fluorescently labeled proteins shown in Fig. 9. As illustrated in Fig. 9, due to the flexible nature of our stencil, one can quite readily pattern curved surfaces.

6.2. *High pattern resolution and pattern flexibility*

To characterize the properties of the parylene-C shadow mask, several parylene stencils with various dimensions, spacings and shapes were fabricated. After fabrication, these membranes were placed over silicon wafers and metal films (Al and Cr–Au) with 1500 Å in thickness were sputter deposited. After the deposition, we have carefully peeled off the shadow mask from the silicon wafer and reused it multiple times without any difficulty. Due to the relatively large dimensions of the features (4–5 μm), one can reuse this mask many times as the holes do not get clogged up and the micropatterns were formed in a reproducible manner. As seen in Fig. 10(a), we were able to achieve fine features as small as 4 μm in a reproducible manner using a 10 μm thick stencil. Utilizing the same shadow mask, we also demonstrated patterning both large and small features simultaneously as displayed in Fig. 10(b) illustrating the pattern resolution of our technology. The feature size measurements that were conducted using both scanning electron microscope (SEM) and the optical microscope agree well with each other and suggest that there is little pattern degradation or blurring during deposition indicating an exceptionally good seal between the parylene film and the silicon substrate.

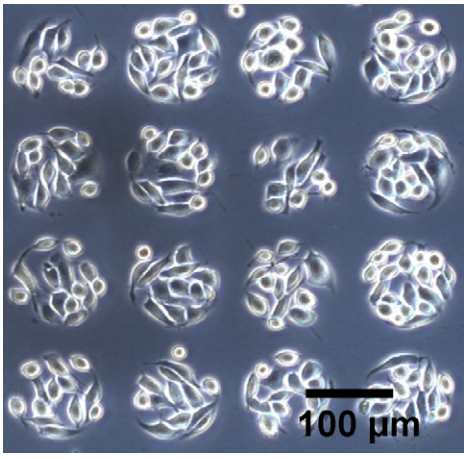

Fig. 8. Patterned NIH-3T3 fibroblast cells.

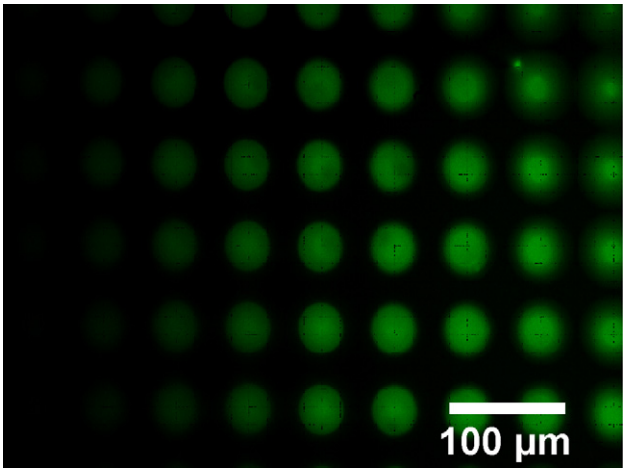

Fig. 9. Images of fluorescently labeled proteins patterned on a curved PDMS surface.

We next demonstrated pattern flexibility using our parylene shadow mask technology as illustrated in Fig. 11. We have successfully patterned structures with different shapes and dimensions. Comparing the parylene shadow masks of thickness 10 μm and 20 μm, we have discovered that the utilization of the 10 μm thick stencil will result in a gap between the stencil and the substrate (due to crimpled surface) when brought in contact with the silicon wafer, hence resulting in deformed patterns. Moreover, the 20 μm thick membrane was rigid enough so that we were able to achieve precise pattern definition as seen in Fig. 11(a–d).

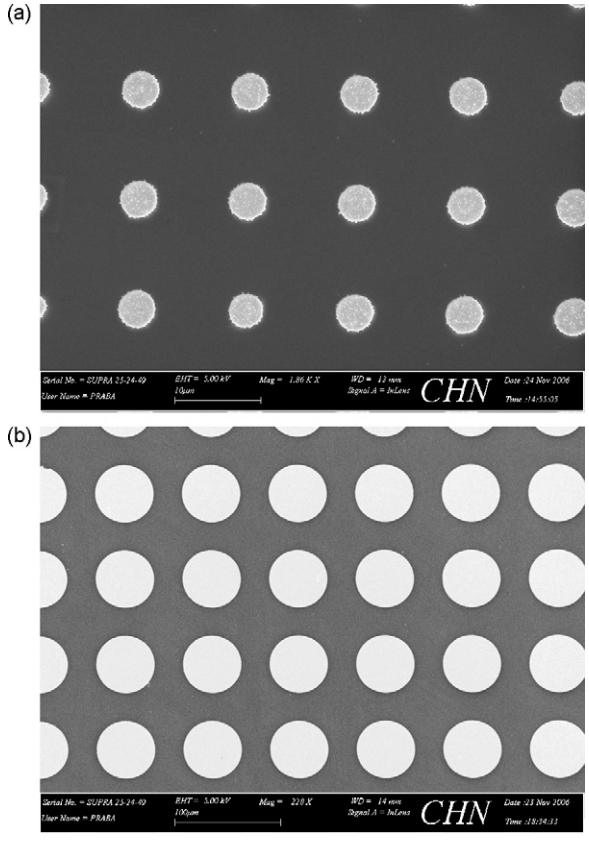

Fig. 10. High pattern resolution achieved using parylene shadow mask. Circular features are patterned with dimensions of (a) 4 μm and (b) 50 μm.

6.3. Micropatterning on curved surfaces

Patterning on curved surfaces has potential applications in flexible electronics and biotechnology. To demonstrate patterning on curved surfaces, we have fabricated a PDMS cylinder which is 17 mm in height and 15 mm in diameter. A 10 μm thick parylene-C shadow mask was subsequently wrapped around this cylinder. Then a 1500 Å thick aluminum film was sputter deposited onto the cylinder and then the parylene shadow mask was peeled off and the resulting micropatterns were imaged using an SEM. Fig. 12(a) displays the optical photograph of the PDMS cylinder with the patterns and Fig. 12(b) displays the magnified SEM micrograph of one of these patterns. Due to the hydrophobic nature of the PDMS surface, the parylene sheet adhered well to the cylindrical surface and the patterns were well defined.

6.4. Mechanical alignment

Most of the current shadow mask technologies are limited to single step patterning applications, yet the benefits achievable from being able to pattern multiple times utilizing alignment features are numerous such as being able to do post processing on released/suspended MEMS devices and in fabricating organic transistors. To carry out a multimask–patterning task, mechanical alignment structures are required. Accordingly, we have designed and fabricated SU-8 alignment posts to hold the parylene shadow masks in place and to align subsequent stencil layers [10]. SU-8 is a fairly thick (up to 500 μm) polymeric material that is being increasingly used in the MEMS and microfabrication fields. Similar to the LIGA process, one can create high aspect ratio structures using a single step exposure.

To create the alignment posts, SU-8-2100 (a negative photoresist, MicroChem Corporation, Newton, MA) is first spun on a 3″ silicon wafer followed by exposure and development. The complete multimask processing sequence is detailed in Fig. 13. We have created 250 μm thick alignment posts made of SU-8 (Fig. 13(a)) to house the shadow masks for subsequent alignment tasks. The first parylene stencil (20 μm thick) was carefully placed inside these posts (Fig. 13(b)). The alignment was verified and adjusted manually under an optical microscope with the fine alignment being performed utilizing a fine tip tweezer. A metal film (Al or Cr–Au with 1500 Å thickness) was next deposited onto the wafer. We have utilized sputter deposition to demonstrate our technique, yet one can also perform any low temperature deposition processes. After removing the first parylene stencil from the silicon wafer, a second parylene stencil with complementary patterns was carefully placed inside the alignment posts (Fig. 13(c)). The second metal deposition was then performed (Al and/or Cr–Au with 1500 Å thickness) and the parylene stencil was subsequently removed from the wafer (Fig. 13(d)). The misalignment from the multimask processing was then examined both under a microscope and under a SEM. To characterize the alignment accuracy in the x and y

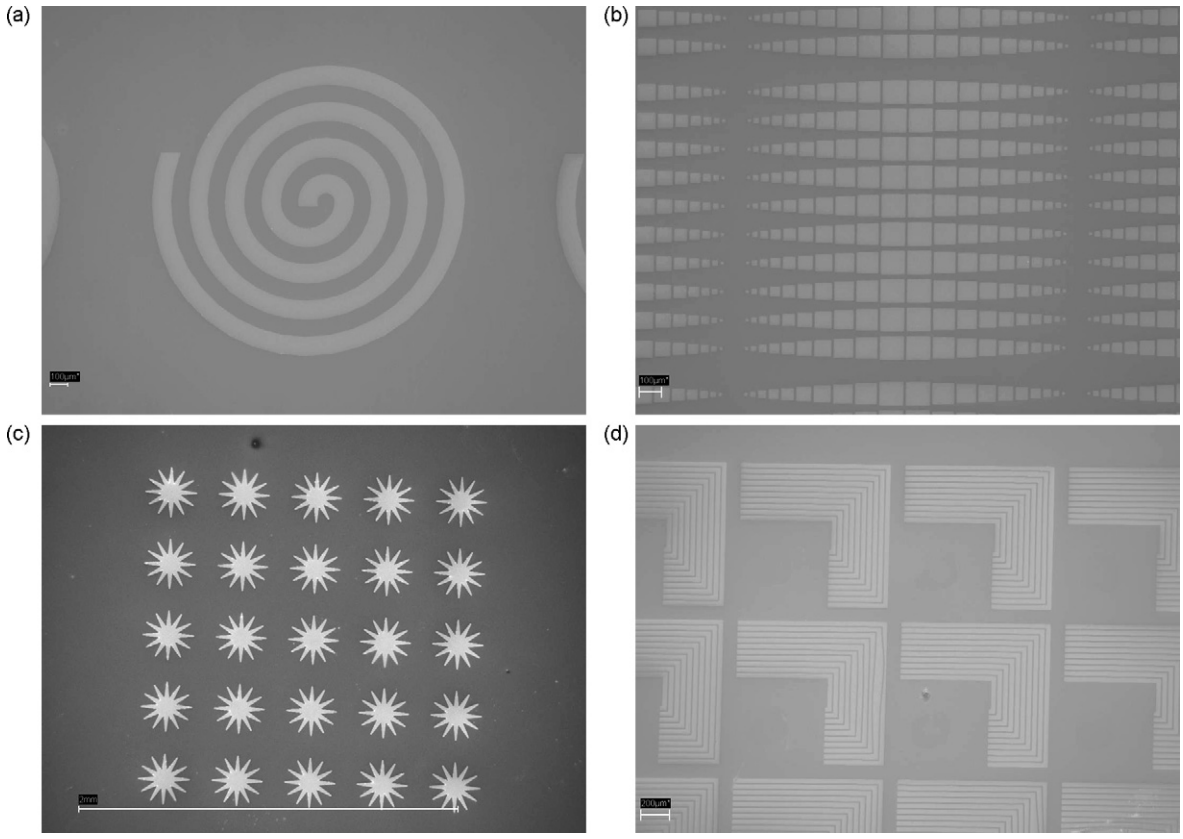

Fig. 11. High pattern flexibility: (a) the width of the spiral is 100 μm, (b) the smallest and the biggest squares are 10 μm × 10 μm and 100 μm × 100 μm and the spacing between squares is 15 μm, (c) starfish patterns, (d) the rectangular patterns where the spacing between lines is 15 μm and the width of an individual line is 25 μm.

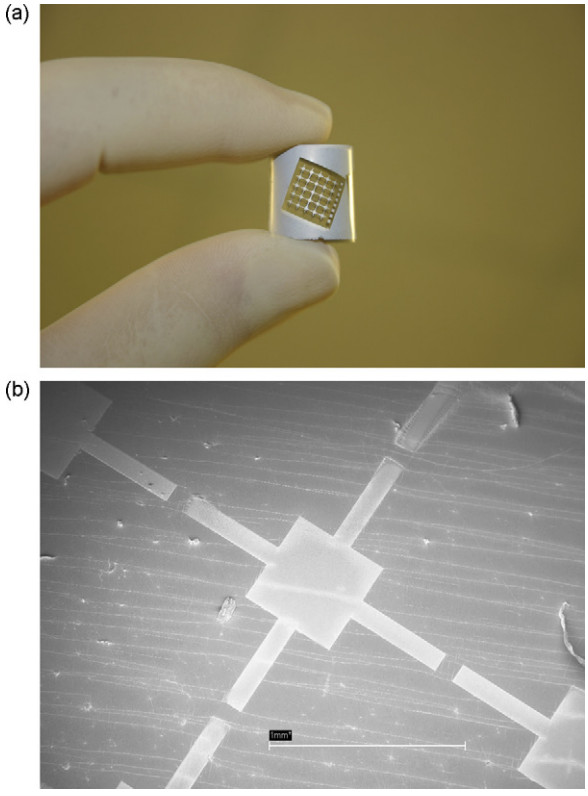

Fig. 12. Micropatterning on curved surfaces: (a) optical image of the PDMS cylinder with micropatterns and (b) magnified SEM micrograph of one of the patterns.

directions, we have created two different complementary "E-shaped" structures [10,19] as seen in Fig. 14. Fig. 15 displays the measurements from the alignment tests. In the *x*-direction, an *x*-offset of 4.6 μm and *y*-offset of 8.6 μm were measured using the complementary structures. In the *y*-direction, the *x*-offset and *y*-offset were 6.9 μm and 4.1 μm, respectively.

In summary, we demonstrated a multistep patterning process with a misalignment of about 4–9 μm using SU-8 pillars. During these experiments, we have utilized two different thicknesses for the SU-8 posts (100 μm and 250 μm). We have found out that the 100 μm posts did not hold the parylene shadow masks properly

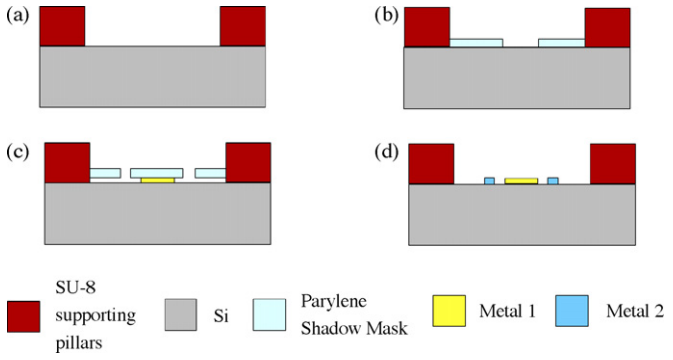

Fig. 13. Micropatterning using SU-8 alignment pillars: (a) fabricate 250 μm thick SU-8 pillars, (b) place the first shadow mask, (c) deposit metal and then peel off the first shadow mask and place the second shadow mask, and (d) peel off the second shadow mask.

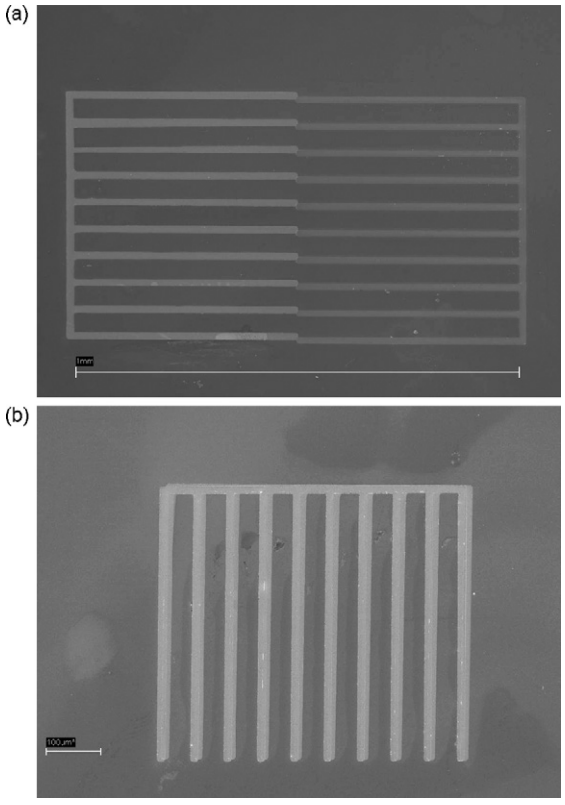

Fig. 14. Test structures for misalignment measurements in x and y directions where the width of the electrode is 10 μm and the spacing between them is 50 μm: (a) alignment tests in x-direction using complementary 'E' shaped structures and (b) alignment test in y-direction using the same structures.

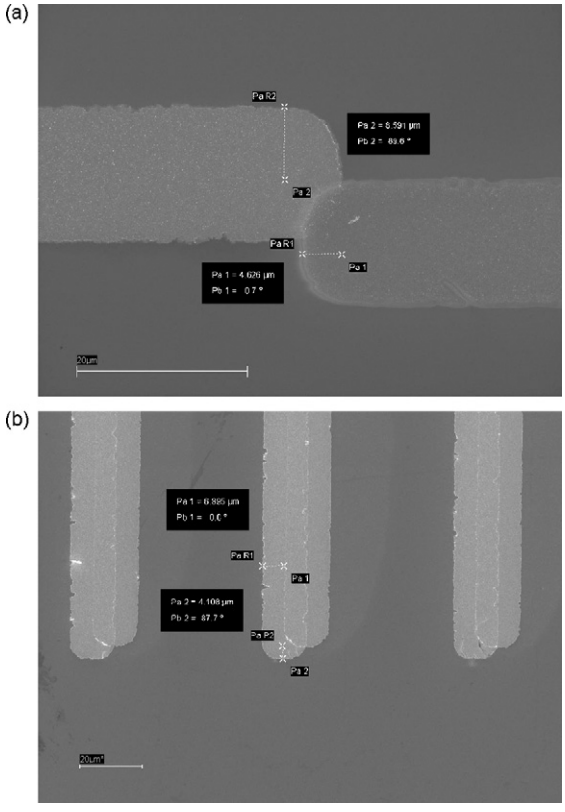

Fig. 15. Measured offsets from the misalignment measurements in x (a) and y (b) directions.

during manual manipulation (for minor adjustments) whereas the thicker version (250 μm) holds the shadow mask in place and hence was the alignment post of choice.

Among its many advantages, parylene shadow masks can be cleaned and are resuable (for at least 10 times) since parylene is an inert material [20]. The fabrication technology is fairly simple, even though we demonstrated our stencils on three-inch wafers, one can easily extend this technology to larger dimensions. Furthermore, due to its mechanical strength and its reusability, one can utilize this technology for patterning large area devices in Flextronics and Macroelectronics as well as patterning for heterogeneous device integration. A final note is that a thin parylene membrane (<10 μm) has the tendency to fold while being held by tweezers, which makes it difficult to precisely position the shadow mask on the sample. Thus, a thicker membrane of at least 20 μm is required for high pattern flexibility and for large area patterning applications.

7. Conclusions

In this paper, we present a flexible, reusable, biocompatible parylene-C shadow mask technology. The minimum feature size of 4 μm is demonstrated while using a 10 μm thick parylene-C stencil for small area patterning applications. A low temperature (5 °C) high aspect ratio (>8:1) parylene etch process was also developed to fabricate the fine structures with anisotropic profiles. Utilizing this flexible shadow mask technology, we demonstrated patterning of proteins and cells on polystyrene, glass and PDMS surfaces. The parylene shadow mask is biocompatible, chemically inert and reusable. Micropatterning of proteins as well as inorganic materials (metals) on curved PDMS surfaces are also demonstrated. Multimask processing is demonstrated with the addition of SU-8 support pillars and the misalignment between masks was measured to be between 4 μm and 9 μm. The parylene stencil method has high pattern flexibility as various shapes with different dimensions can be created utilizing the same stencil. The thickness of the parylene stencil is critical for large area micropatterning applications where a 20 μm thick stencil needs to be utilized. The parylene-C shadow mask technology is versatile and will potentially find diverse patterning applications in numerous fields including organic electronics, macroelectronics, metamaterials and biotechnology.

Acknowledgments

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