

FABRICATION OF FLEXIBLE POLYMER BIO-MEMS WITH SUBMICRON FEATURES

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ABSTRACT

We present a fabrication method for thin-film polymer MEMS that achieves an *order of magnitude* improvement in feature size and resolution. Specifically, we modified the electron beam lithography (EBL) process for biocompatible poly(chloro-para-xylylene) (Parylene C), enabling flexible, encapsulated titanium structures with features as small as 100 nm. The mechanical, electrical and material properties of these Parylene nanodevices were investigated, and strategies to further reduce feature size and achieve batch scale nanofabrication are discussed.

INTRODUCTION

The proliferation of polymer-based MEMS and microelectronics has created a new platform for implantable biomedical devices. Common MEMS materials such as silicon and glass are rigid and therefore poorly suited for chronic implantation in soft tissues [1]; high Young's moduli materials are likely to induce tissue damage and consequentially trigger the body's immune response, leading to the encapsulation or impediment of the device by scar tissue. By adapting standard microfabrication techniques to substrates of biocompatible polymers, researchers can create flexible, 'soft' implants which retain the small size and precise engineering of MEMS technologies. These include electrode array interfaces to the cortex [2, 3], hippocampus [4], peripheral nerves [5], and retina [6, 7].

Many such devices conform to a common archetype: surface micromachined structures fully encapsulated in biocompatible polymer (e.g. Parylene, polyimide, PDMS). Unfortunately, achievable resolution ($> 5 \mu\text{m}$) is constrained by micropatterning techniques compatible with polymer substrates. This restriction precludes polymer-based implants with the component density of silicon devices; for example, silicon neural probe arrays can contain > 100 recording sites [8], while polymer devices typically have merely 4-16 [9]. However, submicron lithography on polymer substrates is non-trivial; many nanopatterning techniques are incompatible with polymers which cannot tolerate high-temperatures, high-intensity radiation, and harsh chemical treatments. In addition, devices intended as flexible implants require highly conformal insulation and robust adhesion of patterned structures to the polymer base, requirements strained as components approach the nanoscale.

Only recently have submicron fabrication techniques, ubiquitous among silicon processing, been applied to polymer substrates [10]. Recently we demonstrated an EBL method for patterning free-film Parylene C devices, with submicron electrical structures [11]. Here we describe an improved method that enables flexible bio-MEMS with features as small as 100 nm, in addition to mechanical and electrical characterization of the resulting

devices. Additionally, we outline a path towards parallelization of this method using plasma-enhanced nano-contact printing on Parylene substrates.

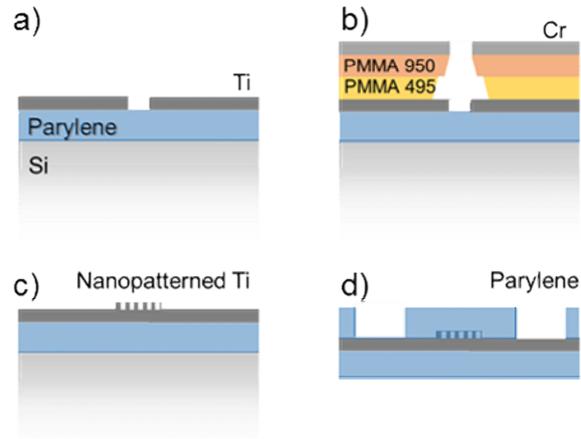


Figure 1 Process flow for submicron fabrication on Parylene C by EBL; (a) Contact pads and traces are fabricated on CVD Parylene, (b) PMMA bilayer is deposited then coated with Cr, and finally patterned by EBL, (c) Ti features are defined by lift-off, (d) Features are encapsulated in Parylene, and the device is released with a O_2 plasma etch.

METHODS

Electron Beam Lithography on Parylene C

A modified EBL method (Fig. 1) was used in conjunction with lift-off to pattern metal structures on Parylene substrates. All experiments used clean silicon dies ($\sim 1\text{-}2''$ sides) coated in $10 \mu\text{m}$ of Parylene C by way of room-temperature chemical vapor deposition (CVD); Silicon served purely as a mechanical support for the Parylene base. A resist bilayer was prepared from polymethylmethacrylate (PMMA, MW: 495k & 950k) dispersed in anisole. Samples were prepared with resist layers either 570 nm (400 nm 495k, 170 nm 950k) or 375 nm thick (300 nm 495k, 75 nm 950k). Each layer was deposited by way of spin-coating, and the samples subsequently baked at $120 \text{ }^\circ\text{C}$ for 20 minutes to drive off residual solvents. Use of higher temperatures recommended by the manufacturer resulted in stress-induced cracking of the Parylene film. Samples were finally coated in 15 nm Cr to offset charge accumulation during e-beam exposure. Features were written at accelerating voltages of $10\text{-}20 \text{ kV}$, exposure dosage of $177 - 600 \mu\text{C}/\text{cm}^2$, with a $30 \mu\text{m}$ diameter aperture. The Cr layer was etched away using Cr-etchant, and the features developed in a solution of methyl-isobutyl ketone in isopropanol (1:3 mixture ratio) for approximately 20 seconds. Features were metallized by evaporating Ti (150 nm thick), followed by lift-off in acetone. Typically devices were then encapsulated in an insulating Parylene C layer ($10 \mu\text{m}$).

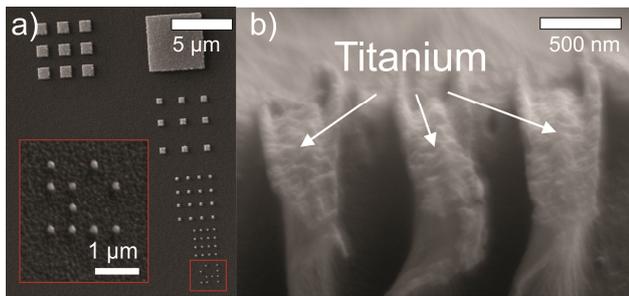


Figure 2 (a) SEM micrograph of Ti squares of decreasing size (5 - 0.10 μm), inset shows enlarged view of 100 nm features. (b) SEM micrograph showing the bisected cross-section of Ti wires (400 nm widths and spacing) encapsulated in Parylene C. Intercalary insulation of Parylene C is clearly visible.

Test Structure Fabrication

Three sets of samples were produced to assess the fabrication technique and the properties of the resulting devices. **First:** Squares with sides ranging 0.075 - 5 μm were patterned on Parylene C films using the above EBL method, and metallized for imaging with scanning electron microscopy (SEM). These samples were used to measure the minimum resolvable pattern the technique could produce, and the smallest achievable features which would adhere to the flexible substrate without delamination. **Second:** Rows of parallel metal wires with spacing and widths 0.04 - 5 μm were fabricated through EBL, encapsulated in Parylene C, then bisected with O_2 plasma. Cross-sections of these samples were imaged by SEM to examine the conformality of CVD Parylene at the submicron scale. **Third:** Conductive test structures were fabricated incorporating straight and serpentine metal traces with widths and spacing of 0.15 - 5 μm and length of 100 μm that spanned larger metal contact pads. Fabrication entailed patterning Ti alignment marks and contact pads on a 10 μm thick Parylene C layer using standard UV lithograph and lift-off, followed by definition of submicron structures using EBL. Devices were encapsulated in Parylene (10 μm), and the contact pads exposed using a deep reactive O_2 etch (DRIE). A second DRIE step cut-out the devices, which were peeled off the silicon carrier.

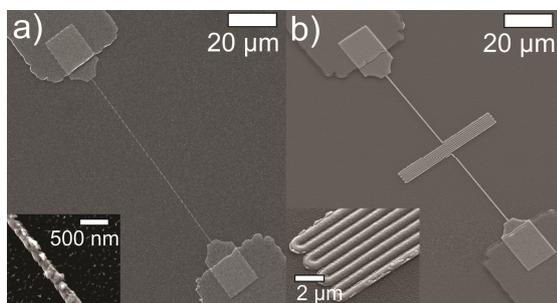


Figure 3 Submicron Ti structures on Parylene C (a) SEM image of straight 0.15 μm trace and (d) serpentine 0.4 μm trace with 0.4 μm spacing.

Characterization & Imaging

Samples were coated with a thin-layer (<5 nm) of Pt, prior to imaging with SEM, to prevent charge build-up in the non-conductive Parylene film. Test structures were probed for electrical DC resistance using a basic 2-point

probe. Resistance measurements were also collected during mechanical testing: electrical contacts were made using wire and conductive silver epoxy, and resistance was recorded during manual deformation, bending, and torquing of the test devices. Devices were also flexed using a moving stage and stepper motor, while measuring change in DC resistance as a function of bend angle.

High-Density Neural Probe Prototype

A prototype flexible neural microprobe was fabricated using a combination of polymer micromachining and the above EBL method. The probe consists of a flexible Parylene shank (2 mm \times 150 μm) supporting 32 Ti electrodes (30 μm diameter). Electrode sites were fabricated on a 10 μm thick Parylene base layer using UV lithography and lift-off. EBL patterning was then used to define the connective traces (750 nm width and spacing, 2 mm long), the critical enabling feature of the device, and also to pattern the electrode surfaces with arrays of raised Ti structures (400 nm width). Finally the outline of the device was defined with DRIE and the device was peeled from the silicon carrier. A similar approach was also attempted to produce a Parylene-based microprobe with 64 electrode sites along a shank of similar dimensions, with more densely spaced connective traces (400 nm spacing and width).

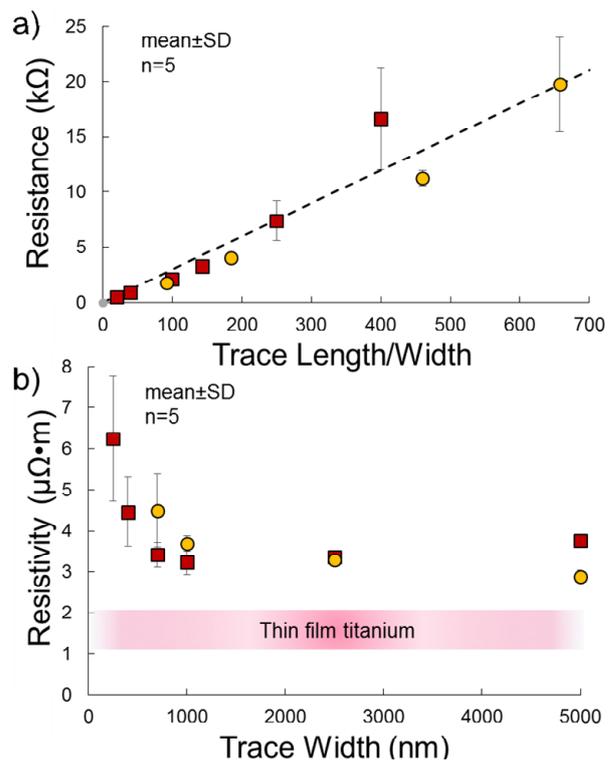


Figure 4 (a) Plot of resistance vs. length-to-width ratio of patterned test structures and (c) plot of resistivity vs trace width. Shaded region spans typical resistivity for thin-film Ti. Straight (square) and serpentine (circle) traces.

RESULTS

Patterns with features as small as 100 nm and as large as 5000 nm were lithographically defined on Parylene substrates through EBL (Fig. 2a). This resolution was achieved with exposure parameters of 420 $\mu\text{C}/\text{cm}^2$ at 20 kV, using the 'thin' (375 nm) resist bilayer. Reducing the

resist thickness allowed for resolution to be improved twofold compared with the thicker (570 nm) resist, while maintaining suitable liftoff profiles. Exposure intensity above $600 \mu\text{C}/\text{cm}^2$ produced distorted patterns and, in some cases, ablated the underlying Parylene, while intensity below $240 \mu\text{C}/\text{cm}^2$ was insufficient to develop patterns. Ti structures with critical dimensions as small as 250 nm could be reliably machined, and exhibited strong adhesion to Parylene even under repeated deformation.

Parylene insulation was remarkably conformal despite submicron length-scales. Figure 2b shows representative SEM images of bisected titanium wires embedded in Parylene C. The intercalary space between a set of structures is fully encapsulated, despite the small pitch and spacing (400 nm). Smaller structures proved difficult to image with clarity, though there was no indication of poor insulation. This result bodes well for the use of such devices in implantable bioMEMS, where good barrier properties is a necessity to minimize water intrusion or electrical crosstalk.

Flexible conducting traces were successfully and reliably produced with submicron widths (0.25-5 μm) and spacing (0.4-5 μm); traces as thin as 100 nm were produced though with considerably lower yield. Representative devices are shown in Figure 3. Linear traces thinner than 250 nm were observed to break along their length, while serpentine traces with spacing closer than 400 nm were frequently distorted due to proximity effects during exposure. Devices were metallized using Ti, Au and Pt, with Ti selected for experiments owing to strong Parylene adhesion and proven biocompatibility. Submicron structures produced with this method were flexible and robust; Ohmic characteristics (Fig. 4a) with low resistivity (patterned Ti: 3.5-6.4 $\mu\Omega\cdot\text{m}$, Fig. 4b) and low contact resistances (50-100 Ω) were observed. Resistivity was notably higher than unpatterned Ti films evaporated to the same thickness (1-2 $\mu\Omega\cdot\text{m}$) [12], and increased with decreasing width.

Resistance varied immeasurably during manually handling, torsion and bending. Data collected using a stepper motor revealed $< 0.5\%$ change in resistance during bending beyond an angle of 75° , and to a radius of curvature below 1 mm (Fig. 5). Sudden and catastrophic failure was observed at bending radii $< 0.2 - 0.3$ mm.

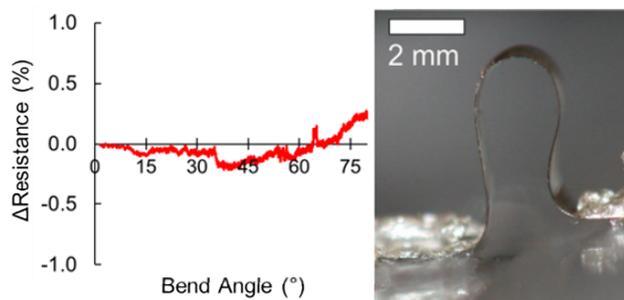


Figure 5 Representative data from mechanical characterization (700 nm serpentine trace): (left) fractional change in resistance plotted against bend angle; (right) side image of bent device during test

A prototype neural microprobe was fabricated (Fig. 6) consisting of a flexible Parylene shank ($2 \text{ mm} \times 150 \mu\text{m}$) supporting 32 electrodes: the highest density array on

a single polymer shank to the authors' knowledge. Connective traces (750 nm pitch and width) were continuous without short or break over their 2 mm length, and exhibited the same flexibility as the conductive test structures. Line resistance was not recorded directly, but can be extrapolated as $\sim 150 \text{ k}\Omega$ DC using the results from electrical characterization. The raised structures patterned on electrode recording sites increased the effective surface area of the electrodes by 40%, compensating for the comparably small geometric surface area of the electrodes ($\sim 700 \mu\text{m}^2$). Fig. 6b shows connective traces from a failed 64-electrode Parylene probe with 400 nm pitch and width. These devices suffered from several shorts and breaks in connective traces due to large proximity effects during e-beam exposure.

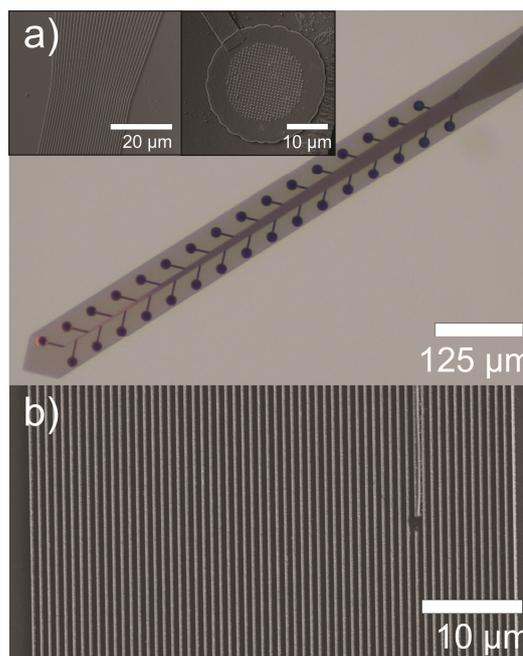


Figure 6 Optical micrograph of prototype polymer neural probe; (a) complete prototype with 32 electrodes: insets show SEM micrograph of (left) 32 connective traces with 750 nm pitch and spacing and (right) nanopatterned electrode site. (b) Section of failed prototype featuring 64 connective traces with 400 nm pitch and spacing, featuring prominent trace break.

DISCUSSION

The high electrical resistivity and low thermal budget intrinsic to polymers creates several obstacles for traditional EBL processing. By preparing e-beam resists at lower temperatures, incorporating a conductive Cr overcoat, and carefully calibrating exposure dose and voltage, EBL was successful on Parylene C substrates. We achieved pattern resolution an order of magnitude greater than that of typical polymer micromachining with high fidelity and good repeatability. Thin-film metal test-structures patterned on Parylene C using EBL were electrically and mechanically robust down to 250 nm, able to withstand the deformation and strain expected of flexible devices without mechanical failure or apparent delamination. Conformality tests were similarly promising, with CVD insulation effectively coating submicron structures without voids despite submicron

intercalary spacing.

Though EBL offers high resolution the process is slow and serial; for example, each high-density neural probe required 90 minutes of write-time per probe in addition to arduous pattern alignment. Outside of small batch production used in research and prototyping, a more time efficient approach is necessary to achieve a practical batch-scale alternative. In on-going work, we are investigating a large-area pattern-transfer technique adapted from nano-contact printing processes used previously with other polymers [13]. The method is briefly outlined in Figure 7. First, a negative of the desired pattern is etched into a glass or oxidized silicon wafer which is coated in Au and a thin Ti adhesion layer. Then the coated wafer and a Parylene coated sample are exposed briefly to air plasma (20% O₂, 80% N₂) which results in exposed hydroxyl surface sites. The two surfaces are pressed together and heated above 100 °C. The Ti layer forms a strong bond with the Parylene surface, and transfers the weakly bound Au pattern to the Parylene film. Preliminary results suggest transfer of high-resolution patterns across large-areas is possible with additional process optimization.

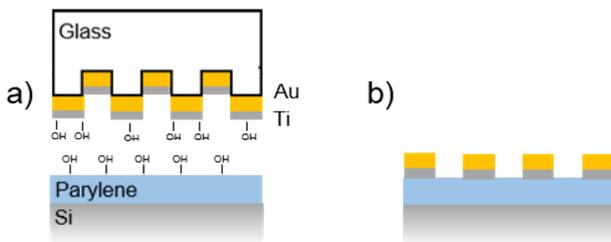


Figure 7 Illustration of nanocontact printing; (a) etched glass stamp is coated in Au/Ti and plasma treated to create hydroxyl sites; (b) metal pattern is transferred to Parylene with application of pressure and heat.

CONCLUSION

Electron beam technology is viable technique for patterning organic and biocompatible polymers with modifications that account for polymer properties. Here we have shown pattern resolution of 100 nm is achievable using thin resist layers and careful calibration of the EBL recipe for use with Parylene substrates. Thin-film structures produced with this method, including electrical components, are electrically and mechanically robust despite the use of a compliant substrate. The combination of EBL with CVD Parylene C enables the creation of flexible bioMEMS with encapsulated submicron components. This approach has applications for novel polymer implantable devices, demonstrated here through the production of a polymer neural microprobe with high electrode density. Submicron processing is a necessary advance for polymer MEMS, and future work will focus on improvements in scale and parallelization.

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