RAPID NON-LITHOGRAPHY BASED FABRICATION PROCESS AND CHARACTERIZATION OF PARYLENE C BELLOWS FOR APPLICATIONS IN MEMS ELECTROCHEMICAL ACTUATORS

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ABSTRACT

We present a rapid (1 day), modular, high-yield (~90%) fabrication process for Parylene C bellows and their mechanical characterization. Load-deflection testing was performed on bellows of varying convolution numbers (1.0, 2.0, and 3.0) and compared to both finite element modeling (FEM) simulations and an analytical model based on membrane deflection theory. Bellows produced a consistent load response. Actuators (consisting of electrodes, electrolyte, and bellows) were assembled and then integrated into а polydimethylsiloxane (PDMS, or silicone rubber) drug reservoir. Preliminary results indicate accurate (< 5% error) drug delivery during repeated dosing at constant flow rate (3.75 µL/min, 2.0 convolution bellows, 1 mA constant current).

KEYWORDS

Parylene C, bellows, electrochemical, actuator

INTRODUCTION

A bellows is a thin-walled corrugated tube [1] that is typically found in flexible coupling elements, in pressure switches or gauges, or as a hermetic housing [2]. In MEMS, bellows have been used in fuel cells [3], endoscopic pressure sensors [4], and microfluidic channel connectors [5].

Bellows can achieve higher deflection with less applied pressure than corrugated or flat diaphragms, as discussed in [6]. Furthermore, additional convolutions can increase the achievable deflection without significantly increasing device size.

However, despite these advantages, bellows are difficult to fabricate using traditional layer-by-layer photolithography-based fabrication processes. Multiple lithography, deposition, and patterning steps are required to lay down the sacrificial and structural materials required to form the bellows structure; multiple photomasks are also required to define the inner and outer diameters of the rings forming the bellows wall [7]. A standard profile of a bellows is shown in Figure 1, where L is the length of one convolution, t the wall thickness, ID the inner diameter of the bellows, OD the outer diameter of the bellows, and H the height of one layer, or half the convolution length.



Figure 1: Standard profile of a bellows.

A sacrificial wax molding technique for Parylene bellows was described but required acid or solvent and elevated temperatures, which induced residual stress in the Parylene [5]. Focused-ion-beam chemical vapor deposition was utilized to fabricate bellows, but was only achieved in carbon film nanostructures and required expensive equipment [8].

Previously, we reported MEMS electrochemicallydriven bellows actuators for drug delivery. The actuator consisted of a Parylene bellows (1.5 convolution only) filled with electrolyte (water) and attached to a platinum interdigitated electrode [6,9]. When activated, the actuator pumped drug out of an adjacent reservoir. Here, we report a new high-yield fabrication process, characterization of bellows having different numbers of convolutions, and preliminary demonstration of accurate dosing using assembled bellows actuators intended for drug delivery applications. Our new non-lithography based process reduces fabrication time (1 week down to 1 day), features reusable molds, and achieved higher yields (~90%) in contrast to our previous work [6].

DESIGN AND FABRICATION

The inner and outer bellows diameters were chosen to correspond with the active area of the electrode. The number of convolutions was varied to determine the potential benefit of additional convolutions to the maximum displacement volume, but was kept low to minimize the overall bellows height in consideration of minimizing dimensions of the drug delivery device. The fabrication process is briefly summarized.

Bellows

Perforated polydimethylsiloxane (Sylgard 184; Dow Corning Corp., Midland, MI) sheets formed reusable molds and were filled with molten (50 °C) low molecular weight (M_n 1000) polyethylene glycol (PEG; Alfa Aesar, Ward Hill, MA). Each PDMS sheet measured 0.40 ± 0.02 mm in thickness. Smoother and less brittle replicas were obtained upon cooling and neither vacuuming nor mold reinforcement was necessary as in [6]. Solidified PEG modules, consisting of one or two layers each as shown in Figure 2, were stacked and fused by moistening the opposing faces of the modules to create bellows molds in increments of 1.0 convolution. Bellows molds (1.0, 2.0, 3.0 convolutions) were coated with Parylene C (9.5 or 13.5 µm; Specialty Coating Systems, Indianapolis, IN), after which the sacrificial PEG was removed by soaking in room temperature deionized water. This new improved bellows fabrication process takes only 1 day.



Figure 2: Bellows fabrication process. Three modules of PEG-filled, 0.4 mm thick PDMS molding sheets with punched 6 (bellows inner diameter) and 9 (bellows outer diameter) mm holes were used in various combinations to rapidly form any desired number of convolutions, and then acted as a sacrificial mold for Parylene C coating.

Electrochemical Actuators

Electrochemical actuators were assembled (platinum electrodes, electrolyte, bellows) using double-sided pressure sensitive adhesive film (3M[™] Double Coated Tape 415, 3M, St. Paul, MN) and reinforced with epoxy (Devcon 5 Minute Epoxy, Danvers, MA). Wires were attached to the electrodes with silver epoxy (EPO-TEK[®] H20E, Epoxy Technology, Inc., Billerica, MA).

Drug Pump

Acrylic molds were machined with a CNC mill and used for casting biocompatible polydimethylsiloxane (MDX-4-4210, Factor II, Inc., Lakeside, AZ) reservoirs. Molds were cured at 80 °C for one hour. An actuator was then integrated into the reservoir and the integrated device was cured at 80 °C for 30 minutes.

THEORETICAL MODELS

Analytical

In the linear bellows approximation [2], bellows were treated as a series of rigidly connected diaphragms having small displacement (defined as deflections < 30% of plate thickness). The total bellows deflection is the sum of the individual diaphragm deflections. However, thin diaphragms can yield displacement greater than 5 times the thickness and have a cubic characteristic equation in relation to the applied pressure [2]:

$$\delta = \sqrt[3]{\frac{3(1-\mu)}{7-\mu}} \frac{Pa^4}{Eh}$$
(1)

In the case of a Parylene C bellows, δ is the bellows deflection, μ is the Poisson's ratio of Parylene C (0.4 [10]), *a* is the diaphragm radius, *P* is the uniform applied pressure, *E* is the Young's modulus of Parylene C (4.75 GPa [11]), and *h* is the thickness of the Parylene bellows wall. The thin diaphragm characteristic equation was the basis for our nonlinear analytical model and approximates the bellows as a series of rigidly connected, *thin* diaphragms. Each bellows layer, as defined in Figure 1, was treated as a diaphragm and the thin diaphragm equation was applied to each layer in the convolution design to determine the approximate total deflection (for example, 5 diaphragms for a 2.0 convolution bellows).

Finite Element Model Simulations

A three-dimensional finite element model of a bellows with alternating diameters of 9 and 6 mm and a convolution length of 0.8 mm (layer height of 0.4 mm) was developed for linear and nonlinear static simulations (Solidworks Simulation 2010, Dassault Systèmes SolidWorks Corp., Concord, MA). 0 to 0.5 psi (0 to 3.45 kPa) loads were applied and the resulting deflection and von Mises stress values were recorded for bellows having 1, 2, and 3 convolutions. Quarter models were used due to geometric symmetry.

EXPERIMENTAL METHODS Mechanical Characterization

Load deflection testing was performed. A pressurized nitrogen gas cylinder was connected to an electronic pressure regulator controlled using a LabView (National Instruments, Austin, TX) interface. Loads from 0 to 0.5 psi (0 to 3.45 kPa) were applied in discrete steps of 0.05 psi to a bellows mounted in a custom acrylic fixture and deflection of the bellows was recorded using a compound microscope with a 100x objective lens (1 μ m resolution).

Maximum Displacement Volume

A constant current of 5 mA was applied to electrochemical actuators and maintained until bellows failure, which was defined as popping of the bellows.

RESULTS/DISCUSSION

Load-deflection test results were compared to the analytical model and FEM simulations. The analytical model underestimated whereas FEM simulations overestimated deflections achieved experimentally, but both provided more relevant approximations than the linear bellows approximation.

Analytical Model

The underestimation by the analytical model is attributed to the rigidity and clamped edge assumptions, which needs to be revised for these polymer bellows (Fig. 3).



Figure 3: Calculated bellows deflection from the analytical model of the bellows (13.5 μ m thick wall) as a series of stacked diaphragms according to the characteristic thin diaphragm equation (shown at the top) for large deflections.

Finite Element Model Simulations

The resulting curves for linear (results not shown) and nonlinear static FEM deflection simulations exhibited a cubic shape similar to that of the analytical model (Fig. 4), although the maximum deflection was higher overall. The yield stress of Parylene (55.2 MPa according to manufacturer and 59 MPa according to [11]) was not exceeded at 0.5 psi and the highest stresses occurred at the convolution edges. The accuracy of the nonlinear FEM is limited by the availability of viscoelastic material parameters of Parylene C and is a likely explanation of the overestimation of the deflection.



Figure 4: FEM quarter model simulation results of a) deflection and b) von Mises stress (at 0.5 psi) for 1.0, 2.0, and 3.0 convolutions bellows showed that the yield strength (55.2 MPa) of Parylene C was not exceeded and that the bellows were operating within the elastic region. Highest stresses were concentrated at convolution edges.

Load-Deflection Testing

Load deflection results are displayed in Figure 5. No plastic deformation was observed in visual inspection before and after testing for all bellows designs and responses were consistent across multiple load cycles. As expected, bellows wall thickness followed a nonlinear inverse relationship with deflection (Fig. 5b). Minor hysteresis (~30-50 μ m) was observed, but was largely due to high flow resistance in the testing setup (data not shown). Remaining hysteresis is attributed to the viscoelasticity of Parylene [12].



Figure 5: a) Load-deflection results for 1.0, 2.0, and 3.0 convolutions bellows, b) effect of wall thickness on deflection of a 2.0 convolution bellows, c) consistent load response during repeated testing of a 2.0 convolutions bellows when pressure was cycled to 0.5 psi, and d) a 2.0 convolutions, 13.5 μ m thick bellows before and after deflection testing showed no signs of plastic deformation.

Maximum Deliverable Volume

Table 1 shows preliminary results for maximum deliverable volume, which, as expected, increases with the number of convolutions. A leak in the 3.0 convolution bellows may have contributed to a smaller increase going from two to three convolutions and further testing is underway to improve the reliability of the bellows.

Table 1: Maximum Deliverable Volume of Various Convolution Designs

Convolutions	Maximum DeliverableVolume (μ L)
1.0	131
2.0	228
3.0	258*

Flow Testing of Drug Pump

Electrochemical actuators were integrated into a PDMS drug reservoir to form a drug pump and operated at constant current. Preliminary results indicate accurate drug delivery during repeated dosing (n = 4) at constant flow rate (3.75 μ L/min, 2.0 convolution bellows, 1 mA, Fig. 6). Slight variation in the fluid delivered between runs is largely due to the electrolysis phenomena occurring at the electrodes and not the bellows; it is expected that improved repeatability will be obtained with Nafion[®]-coated electrodes [13].



Figure 6: Preliminary results on accurate dosing achieved with bellows integrated into an electrochemical actuator and drug reservoir. a) illustration and photo of actuator structure and b) photo of top view of drug pump and flow testing results.

ACKNOWLEDGEMENTS

This work was supported by a Wallace H. Coulter Foundation Early Career Translational Research Award and a National Science Foundation Graduate Research Fellowship (Gensler). Special thanks to Diya Dwarakanath and Heather Chen for their assistance with the bellows fabrication.

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