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Parylene BioMEMS

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Abstract

This paper describes Parylene as an emerging BioMEMS material. Parylene has the unique feature of room-temperature, pinhole-free conformal CVD deposition. It is chemically inert and biocompatible. More interestingly, it is found that Parylene thin film usually possesses a tensile intrinsic stress, controlled by the last thermal steps. These features allow free-standing parylene MEMS structures in many designs. Parylene MEMS is also a suitable technology for post-CMOS integration. As a result, multi-layer Parylene MEMS technology has been developed, especially for BioMEMS applications. This paper also gives examples of integrated Parylene microfluidics and HPLC on-a-chip.

1. Parylene as a MEMS material

Parylene is the generic name for members of a unique family of thermoplastic polymers that are deposited by using the dimer of para-xylyene (di-para-xylylene, or DPXN). Parylene coating processes were originally developed in the 1950s by William F. Gorham and then commercialized by the Union Carbide Corporation in 1956. Parylene is widely used for coating of electronic components, medical instruments, and has recently been explored as the interlayer dielectrics (ILD) in the microelectronics industry. The main reason for studying Parylene material to be used in MEMS applications lies in its unique room-temperature CVD conformal deposition characteristics. As demonstrated, the Parylene MEMS technology using photoresist as the sacrificial material requires processing temperatures no higher than 120°C, which is a great advantage in terms of cost consideration and. As shown in Fig.1, there are three different kinds of Parylene commercially available on the market. Parylene N is poly-para-xylylene, a completely linear and highly crystalline polymer. Parylene C, which is the main focus of study in this thesis, is basically Parylene N with a chlorine atom replacing one of the aromatic hydrogens. Parylene D is similar to Parylene C but with two aromatic hydrogens being replaced with chlorine atoms. The benzene backbone of the parylenes makes them very *chemically inert*. At room temperature, there is no known chemically that will significantly attack the parylenes.

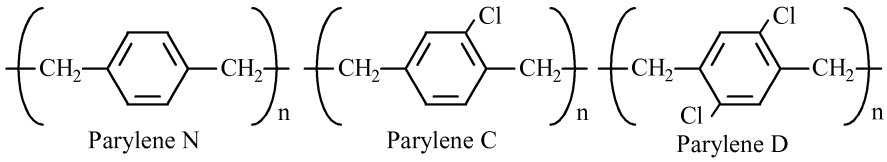

Figure 1 *The three most popular Parylenes.*

Parylene is deposited at room temperature around 0.1 torr where the mean free path of the molecules is on the order of 0.1cm. The first step of the deposition process is the vaporization of the solid dimer at approximately 150°C. The second step is the pyrolysis of the dimer at about 650°C, in which the dimer is cleaved at the two methylene-methylene bonds to yield the stable monomeric diradicals, para-xylylene. Finally, the monomers enter the deposition chamber at room temperature where they polymerize on the substrate. The cold trap at approximately -70°C is used to collect the un-reacted monomers before they enter the mechanical pump. Although Parylene can be deposited selectively using heated resistors to create local heating, Parylene is best patterned with plasma etching, instead of chemical etching. Because Parylene deposition is conformal, lift-off process to pattern the Parylene is difficult. Therefore, O_2 plasma etching with photoresist as the mask is used in most cases. Other metals such as sputtered silicon have been explored as etching masks. However, the most important criteria for selecting a mask material is the adhesion between the etch mask and the material itself. Adhesion of photoresist and Parylene is excellent because they are both polymers, whereas some metals such as evaporated aluminum do not guarantee good adhesion and therefore have to be used

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with great care. A-174 is the adhesion promoter suggested by the manufacturers in order to enhance the adhesion between the Parylene film and many substrates (Liger, Rodger and Tai, 2003). The ease of Parylene process is further demonstrated by a multi-layer Parylene MEMS technology with as many as 5 layers of Parylenes (Wang and Tai, 1999) without special planarization techniques. Mechanically, parylene has a high electrical resistivity and low Young's modulus (2-5 GPa).

2. Biocompatible Parylene for BioMEMS

It is known that materials like silicones (e.g., PDMS) and polyimides could be "short-term" (e.g., for 3 months) biocompatible, but they are not known to be "long-term" biocompatible materials. On the other hand, Parylene is a much better biocompatible as it is an US Pharmacy (USP) officially-approved class-VI implantable plastic material. It has a long history of being used in many biomedical devices such as pacemakers (for 3 decades) and even implantable electronic packages. More importantly, a complete set of useful Parylene MEMS microfluidics devices including valves, pumps, reaction chambers, and even HPLC have been developed for the last 10 years (Tai, 2003). This Parylene microfluidics technology can now be fully available.

3. Integrated Parylene BioMEMS

MEMS can have very interesting microflow control applications (Ho and Tai, 1998), but time may prove that BioMEMS may be the biggest winner. Here, then, some important examples of Parylene microfluidics valves and pumps developed for BioMEMS applications are described.

3.1 In-channel Check Valves

Fig.2 shows the first surface-micromachined, normally closed, in-channel, Parylene check valve (Wang and Tai, 2000). This device is fabricated monolithically on a silicon substrate using a five-layer Parylene process. The operating structure of the check valve is a circular sealing plate on top of a ring-shaped valve seat. The sealing plate is center-anchored on top of a chamber diaphragm that is vacuum-collapsed to the bottom of the chamber in order to achieve a normally closed position. A thin gold layer on the roughened valve seat surface is used to reduce stiction between the sealing plate and the valve seat. We have achieved an in-channel check valve with a cracking (opening) pressure of 20~40 kPa under forward bias and no measurable leakage under reverse bias up to 270 kPa. Using this design, this valve performs well in two-phase microfluidic systems (i.e. microchannel flows containing gas, liquid, or gas/liquid mixture).

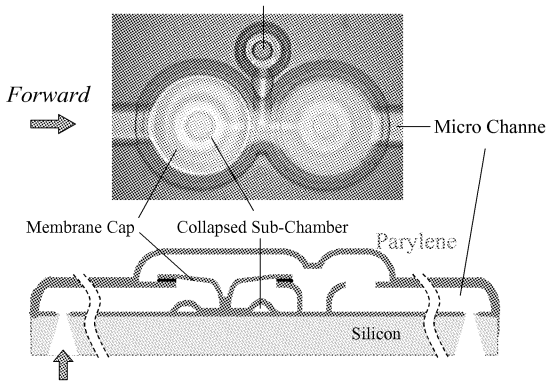

Figure 2 a surface-micromachined normally closed in-channel check valve using 5-layers of Parylene.

3.2 Integrated Mass Flow Controller

An integrated surface-micromachined mass flow controller (MFC) that consists of an electrostatically actuated microvalve and a thermal flow sensor is shown in Fig. 3 (Xie and Tai, 2003). With a special design and a multilayer Parylene process, the active microvalve and the flow sensor are integrated onto a single chip to perform closed-loop flow control. Sensitivity of the flow sensor is 55 μV/(μL/min) for air flow and 12.2 μV/(nL/min) for water. Valve is actuated with 10 kHz AC voltage and 200 Volts can close the valve while

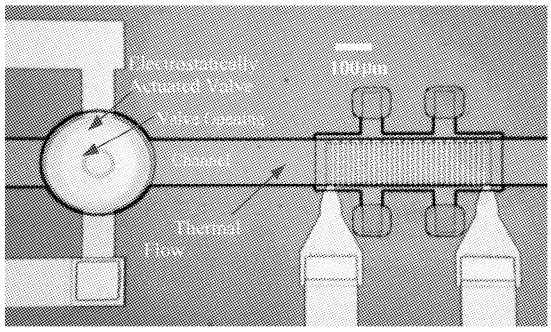

Figure Fabricated mass flow controller

3 psi pressure is applied. For flow control, both Pulse Width Modulation (PWM) and actuation voltage adjustment are used. The testing results show that PWM has better control capability and linearity.

3.3 Electrostatic Parylene Peristaltic Pump

Fig. 4 presents a new 4-Parylene-layer surface-micromachining platform technology and its application for an electrostatic peristaltic micropump (Xie, Shih and Tai, 2004). CVD Parylene is used as the structural material, while normal photoresist is the sacrificial layer and Cr/Au the electrode. This versatile process enables a peristaltic pump design, in which electrical field does not exist inside the fluid. Testing shows an electrostatic pull-in voltage of 150V for a 200-μm-diameter membrane. 3 and 6-phase peristaltic pumping actuations are used and pumping of either water or ethanol is demonstrated. A pumping rate of 2nL/min (flow velocity of 100μm/sec) is achieved at 60Hz for ethanol. Higher flow rate (>10nL/min) is feasible with improved design.

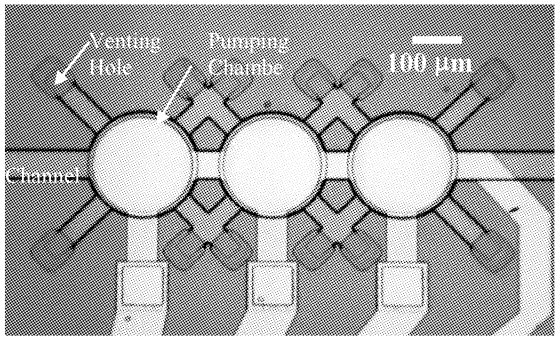

Figure 4 Fabricated micro peristaltic pump.

4 Integrated Chip for Protein Mass Spectral Analysis

Finally, Fig. 5 presents the first fully integrated gradient-elution liquid chromatography-electrospray ionization (LC-ESI) system on a chip (Xie et al, 2005). This chip integrates a pair of high-pressure gradient pumps, a sample injection pump, a passive mixer, a packed separation column, and an ESI nozzle. We also present the successful on-chip separation of protein digests by reverse phase (RP)-LC coupled with on-line mass spectrometer (MS) analysis. Fig. 6 shows an example of BSA peptides that are separation by the HPLC chip. Therefore, we have demonstrated the first fully integrated LC-ESI system on a chip. The chip consists of (gradient and sample injection) pumps, a mixer, a column, filters, and an ESI nozzle to create a functional device. Current work involves integrating sensors for feedback control and improving separation performance.

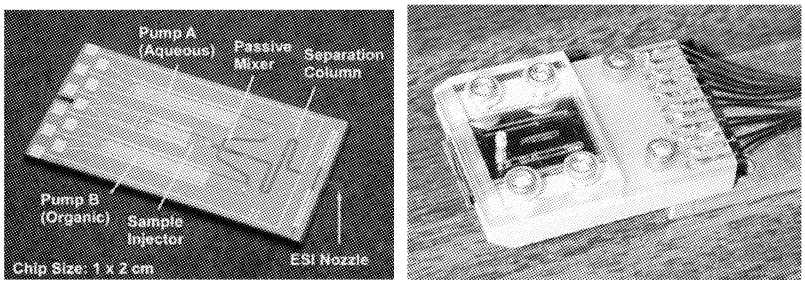

Figure 5 A photograph of the fabricated HPLC lab-on-a-chip. The picture on the right is a fully packaged HPLC system that can be connected directly to mass spectrometers through electrospray ionization (ESI).

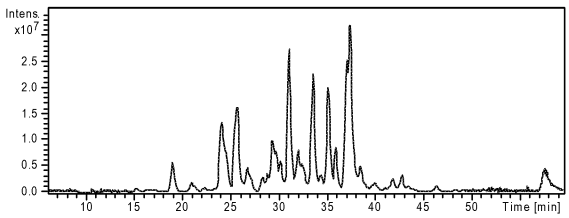

Figure 6 Separation of BSA Digest using commercial HPLC system.

5. Conclusions

This paper describes Parylene as an emerging BioMEMS material. In addition, multi-layer Parylene MEMS technology has also been developed, which enables many important BioMEMS applications. This paper gives examples of integrated microfluidics. Moreover, HPLC on-a-chip is also described as an example of fully integrated and complex BioMEMS system.

Acknowledgements

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