Fabrication of Polydimethylsiloxane (PDMS) - based Multielectrode Array for Neural interface

Jun-Min Kim¹, Da-Rong Oh², Joaquin Sanchez³, Shang-Hyub Kim⁴ and Jong-Mo Seo⁵

Abstract—Flexible multielectrode arrays (MEAs) are being developed with various materials, and polyimide has been widely used due to the conveniece of process. Polyimide is developed in the form of photoresist. And this enable precise and reproducible fabrication. PDMS is another good candidate for MEA base material, but it has poor surface energy and etching property. In this paper, we proposed a better fabrication process that could modify PDMS surface for a long time and open the site of electrode and pad efficiently without PDMS etching.

I. INTRODUCTION

Neural interface technology is one of the hot research issue in these days. Recent development of science and technology gives hope to restore the damaged human sensory function and brain[1]. By enabling interface between human and computer. The most important technical part of artificial sensory or brain computer interface(BCI) is a measurement of the various electrical signals from the body and the development of the electrodes which can deliver electrical stimulation the to nerves[2], [3], [4], [5], [6]. The miniaturization of electrodes and development of biocompatible electrodes are being investigated as the core technology of BCI[7].

In this paper, the fabrication process of planar type electrode in various types has been developed using PDMS as soft material. Through proposed process, problems which is poor adhesion between metal and PDMS, difficulty of PDMS etching and reproducibility in the fabrication process would be improved.

II. METHODOLOGY

A. Summary of Fabrication

PDMS layer is spun on a silicon wafer, and then gold layer was deposited and patterned using wet etching process. Sacrificial posts are formed using negative photoresist on the electrodes and contact pads where openings are to be made. The device is encapsulated with thin second PDMS layer. Then second PDMS is etched about $1\mu m$ by tetrabutylammonium fluoride(TBAF) solution which can etch hardened PDMS[3]. The sacrificial posts are removed to expose the

¹Jun-Min Kim is with Department of Electrical Engineering, Seoul National University, Seoul, Korea junmin83 at gmail.com.

²Da-Rong Oh is with Department of Electrical Engineering, Seoul National University, Seoul, Korea ddyoy88 at gmail.com.

³Joaquin Sanchez is with Department of Electrical Engineering, Seoul National University, Seoul, Korea joaquinsd10 at gmail.com.

⁴Shang-Hyub Kim is with Department of Electrical Engineering, Seoul National University, Seoul, Korea icebug at snu.ac.kr.

⁵Jong-Mo Seo is with Department of Electrical Engineering and Department of Ophthalmology, School of Medicine, Seoul National University, Seoul, Korea callme at snu.ac.kr. electrode and contact pad opening. Outline of device is cut by ND:YAG laser. Finally, the device is harvested from the silicon wafer by peeling-off. Sequential processes for fabricating MEAs are shown schematically in Fig.1 and are described in detail below.

B. Details of Fabrication

A cleaned silicon wafer is coated with trichloro (1H, 1H, 2H, 2H-tridecafluoro-n-octyl) silane in vacuum desiccator to make weak bonding between silicon and PDMS. It is to facilitate device peeling-off in the end (Step 1 in Fig.1).

PDMS (Sylgard[®] 184, Dow Corning) elastomer base is mixed with curing agent at 10:1 weight ratio. The PDMS layer is formed by spin-coating the de-aired PDMS mixture onto the silane coated silicon wafer. The spin speed and duration are 1000 revolution per minute (rpm) for 30 seconds (s). The coated thickness is about $75\mu m$. Then, the sample is cured in oven at $80^{\circ}C$ for 4 hour (Step 2 in Fig.1).

The sample is modified using oxygen plasma and 2hydroxyethyl methacrylate (HEMA, Aldrich chemical) grafting in a reactive ion etching (RIE) system to increase surface energy of cured PDMS[8]. The modication was conducted in several steps. Oxygen modication was carried out at a system pressure of 75mTorr and flow rate 20sccm, and an RF power of 100W was used for 30s for the modication. Then HEMA was spin coated onto the oxygen modified PDMS surface at a spin speed of 1500rpm for 15s. After oxygen plasma modication, the HEMA-coated PDMS was treated with oxygen plasma at a constant pressure of 75mTorr at an oxygen ow rate of 20sccm. RF power was 100W for 60s. By this process, HEMA is polymerise on PDMS film (Step 3 in Fig.1).

Following this treatment, the sample is deposited with a 3000\AA gold layer primed by 300\AA titanium in an electron gun evaporator (ZZS550, MAESTEK) to prepare the gold pattern using wet etching (Step 4 in Fig.1).

Positive photoresist (AZ1512, Az Electronic Materials) is spun on at 4000*rpm* for 35*s* with ramp rate of 500rpm/sand cured on hotplate at $95^{\circ}C$ for 90s. Photoresist layer thickness is $1.2\mu m$. The sample is then patterned with UV exposure energy dose of $144 mJ/cm^2$ at 365nm and soaked in developer (AZ300MIF, Az Electronic Materials) for 60s(Step 5 in Fig.1).

Gold layer is etched by iodine-potassium-iodide solution in normal temperature. Finally, titanium is exposed and etched by buffered oxide etcher (Step 6 in Fig.1).

To setup sacrificial posts on electrode, a thick layer of negative photoresist (DNR-L300, DONGJIN) is spun onto



Fig. 1. Illustration of fabrication process for PDMS-based Electrode.

the sample. Photoresist layer is patterned to leave sacrificial posts on each electrode and contact pad. Photoresist is spun on at 1300rpm for 20s with ramp rate of 500rpm/s and to achieve about $20\mu m$ thick photoresist layer using double spin-coating scheme. The sample is patterned with UV exposure energy dose of $810 \ mJ/cm2$ at 365nm i-Line and immersed in developer for 100s (Step 7 in Fig.1).

A second PDMS layer is spun on to encapsulate the device at 5000rpm for 60s with a ramp rate of 1000rpm/s. At this time, PDMS is diluted by hexane. The ratio of PDMS to hexane is 1:2. The sample is left at room temperature for 1 hour, and then cured in a $80^{\circ}C$ oven for another 1 hour. The encapsulation thickness is about $5\mu m$. To open electrodes and contact pads, the sample is soaked in TBAF solution for 30s. And the sample is rinsed with acetone stream (Step 8 in Fig.1).

The outline of MEAs is cut ND:YAG laser. The complete device is peeled off its silicon wafer[9]. Finally, the device is cleaned by sonic cleaner for 30s (Step 9 in Fig.1).

III. RESULT AND DISCUSSION

There were many hurdles in establishing proposed fabrication process, and the major point are as follows.

A. Trichlorosilane Layer for The Easy Release of PDMS MEA from Silicon Wafer

After cutting the outline using ND:YAG PDMS MEA should be released from silicon wafer with tweezers in Fig.2. In Fig.2(a), PDMS is coated on the wafer that is not coated with silane. In this case, device cannot be peeled off properly. In Fig.2(b), silane weakened adhesion layer between silicon and PDMS, device is peeled off easily.



Fig. 2. Adhesiion test between silicon and PDMS: (a) Without Trichlorosilane layer (b) After treating trichlorosilane layer

B. Gold Layer Deposition on PDMS

PDMS is hydrophobic material. In order to coat photoresist or deposit metal film, hydrophilic surface of PDMS is needed. There are several options to change the surface property. We evaluated oxygen plasma treatment and HEMA treatment. Thickness of gold film is 300nm. Fig.3(a) shows cracks on gold layer when the surface of PDMS is hydrophobis. In Fig.3(b), gold layer build flat when pretreated O_2 plasma. Case of Fig.3(c), owing to HEMA is polymerized in wavy form on PDMS, glod layer also has wavy form. O_2 plasma treatment can keep the surface hydrophilic only for a few hours, but HEMA treatment preserves hydrophilicity for a long time, thus we choose HEMA treatment. [8].

C. Metal Layer Patterning

There are two methods of forming a metal pattern; Lift-off process and dry/wet etching process.

1) lift-off process: Lift-off usually use photoresist as a mask, and the metal is deposited by evaporator. And then, by removing photoresist, pattern is generated. However, when photoresist is in soft bake process, due to difference of heat expansion rate, photoresist is damaged like Fig.4(a). After photoresist layer is removed, there is also a crack on the PDMS layer like Fig.4(b). Therefore, Lift-off method cannot be applied on PDMS layer.



Fig. 3. Adhesion test between gold and PDMS: (a) No surface treatment (b) O_2 plasma treatment (c) HEMA treatment



Fig. 4. Lift-off problem: (a) Photoresist layer crack (b) PDMS layer crack

2) dry/wet etching process: There are two ways to etch metals such as gold, titanium; dry method which use plasma, and wet method which dissolve the specific metal with etchant. Fig.5(a) shows the result of dry etching. There are red residues on PDMS layer after dry etching process. In this case, RIE uses Ar, CH_4 , Cl_2 gases. Even though, we assume that red-colored residue may not affect the performance of electrode. In wet etching, there are no residues in wet etching as shown in Fig.5(b). In this paper, we adopted wet etching process.



Fig. 5. Dry etching problem: (a) Dry etching (b) Wet etching

D. Electrode and Pad Opening

Finally, to open electrodes and pads, thin PDMS coating process is performed. Sacrificial post was built about $20\mu m$ thickness, and thin PDMS layer is added by $5\mu m$. Due to sacrificial post height, PDMS cannot cover the sacrificial post. After PDMS curing, photoresist is removed by acetone stream and if this step is solely effective, then electrodes and pads should not be covered with PDMS[2], [4]. However, as shown in Fig.6(a), thin PDMS residues are found on

electrodes and pads. To solve this problem, the wet etching process is performed using a solution of tetrabutylammonium uoride (TBAF) in n-methyl-2-pyrrolidinone (NMP), 1:3 (v/v) both supplied by Tokyo Chemical Industry[3]. After PDMS wet etching, Fig.6(b) shows no PDMS residues on electrodes and pads.



Fig. 6. Dry etching problem (a) Electrode site (b) Pad site

E. Manufactured MEAs

Through proposed process, PDMS-based MEAs are successfully fabricated as Fig.7(a). Minimum line width is $20\mu m$, and the diameter of electrodes is $250\mu m$. Thickness of the MEAs is about $75\mu m$, and thickness could be easily changed from tens of micrometers to more than one thousand by changing spin-coating speed.

IV. CONCLUSION

The PDMS have good biocompatibility, but low surface energy makes difficulties in the fabrication process. In order to overcome these problems, HEMA process was introduced. There are two conventional schemes to open electrodes and pads. The one is to attach bottom plate and top plate using alignment machine. And the other is to build sacrificial post. The former method has the disadvantage of poor accuracy and reproducibility because of difficulty in alignment. And the latter which build a sacrificial post based on photo process has good reproducibility, low process yield. In this paper, we were able to enhance the yield of the sacrificial post method.

We are evaluating the performances of the electrode array, and also in vitro and in vivo biocompatibility are being investigated.[10]. These electrodes will be used in recording the extracellular local field potential or stimulating nerve plexus as a contact electrode.

ACKNOWLEDGMENT

This paper was supported by the Korea Health 21 R&D Project A050251 by MIHWAF, Technology Innovation Program 10033634 by MKE, and Public Welfare and Safety Program 2012-0006567 by MEST.



Fig. 7. Manufactured MEAs : (a) The whole MEA structure (b),(c) electrode sites (d),(e) Pad opening

References

- C. E. Schmidt and J. B. Leach, "Neural tissue engineering: Strategies for repair and regeneration," *Annual Review of Biomedical Engineering*, vol. 5, pp. 293–347, 2003, pMID: 14527315.
- [2] T. Adrega and S. P. Lacour, "Stretchable gold conductors embedded in pdms and patterned by photolithography: fabrication and electromechanical characterization," *Journal of Micromechanics and Microengineering*, vol. 20, p. 055025, 2010.
- [3] B. Balakrisnan, S. Patil, and E. Smela, "Patterning pdms using a combination of wet and dry etching," *Journal of Micromechanics and Microengineering*, vol. 19, p. 047002, 2009.
- [4] L. Guo and S. DeWeerth, "Implementation of integratable pdmsbased conformable microelectrode arrays using a multilayer wiring interconnect technology," in *Engineering in Medicine and Biology Society*, 2009. *EMBC* 2009. Annual International Conference of the IEEE, sept. 2009, pp. 1619 –1622.
- [5] B.-H. Jo, L. Van Lerberghe, K. Motsegood, and D. Beebe, "Threedimensional micro-channel fabrication in polydimethylsiloxane (pdms) elastomer," *Microelectromechanical Systems, Journal of*, vol. 9, pp. 76 –81, march 2000.
- [6] K. Meacham, R. Giuly, L. Guo, S. Hochman, and S. DeWeerth, "A lithographically-patterned, elastic multi-electrode array for surface stimulation of the spinal cord," *Biomedical Microdevices*, vol. 10, pp. 259–269, 2008.
- [7] A. Jackson, C. Moritz, J. Mavoori, T. Lucas, and E. Fetz, "The neurochip bci: towards a neural prosthesis for upper limb function," *Neural Systems and Rehabilitation Engineering, IEEE Transactions on*, vol. 14, no. 2, pp. 187–190, june 2006.
 [8] D. Bodas and C. Khan-Malek, "Hydrophilization and hydrophobic
- [8] D. Bodas and C. Khan-Malek, "Hydrophilization and hydrophobic recovery of pdms by oxygen plasma and chemical treatment-an sem investigation," *Sensors and Actuators B: Chemical*, vol. 123, pp. 368 – 373, 2007.
- [9] M. Wehner, P. Jacobs, and R. Poprawe, "Rapid prototyping of microfluidic components by laser beam processing," pp. 645 908–645 908– 12, 2007.
- [10] S. F. Cogan, "Neural stimulation and recording electrodes," ANNUAL REVIEW OF BIOMEDICAL ENGINEERING, vol. 10, pp. 275–309, 2008.