OPTOELECTRONICS LETTERS Vol.12 No.3, 1 May 2016

Thick SU8 microstructures prepared by broadband UV lithography and the applications in MEMS devices[*]

LI Dong-ling (李东玲)[**], WEN Zhi-yu (温志渝), SHANG Zheng-guo (尚正国), and SHE Yin (佘引)

National Key Laboratory of Fundamental Science of Novel Micro/Nano Device and System Technology, Key Laboratory of Optoelectronic Technology and System of the Education Ministry of China, Micro System Reaserch Center, Chongqing University, Chongqing 400030, China

(Received 12 January 2016)

Thick SU8 microstructures with high aspect ratio and good side wall quality were fabricated by ultraviolet (UV) lithography, and the processing parameters were comprehensively studied. It proves that the adhesion of SU8 on silicon (Si) substrates is influenced by Si-OH on the surface, and can be improved by the HF treatment. Cracks and delamination are caused by large internal stress during fabrication process, and are significantly influenced by soft bake and post-exposure bake processes. The internal stress is reduced by a low post-exposure bake exposure temperature of 85 °C for 40 min. A three-step soft bake enhances the reflowing of SU8 photoresist, and results in uniform surface and less air bubbles. The vertical side wall is obtained with the optimized exposure dose of 800 mJ/cm^2 for the thickness of 160 μm. Using the optimized fabrication process combined with a proper structure design, dense SU8 micro pillars are achieved with the aspect ratio of 10 and the taper angle of 89.86°. Finally, some possible applications of SU8 in micro-electromechanical system (MEMS) device are developed and demonstrated.

Document code: A **Article ID:** 1673-1905(2016)03-0182-6

DOI 10.1007/s11801-016-6015-4

SU8 is an ultra-thick, negative tone, epoxy-based and near-ultraviolet (UV) (350—400 nm) photoresist, and its low optical absorption in UV range allows high aspect ratio patterning with smooth and near-vertical sidewalls. Additionally, after development, it has a high thermal and chemical stability due to the highly crosslinked epoxy rings of the SU8 molecules[1]. Thick SU8 structures have been widely used in micro-electromechanical system (MEMS) and micro-opto-electromechanical system (MOEMS) devices. For example, SU8 is used as a temporary material for sacrificial etching of hollow waveguides[2], a photolithographic material for etch masks[3], electroplating[4] and wafer bonding layers[5]. SU-8 is also widely used as a structural material to build MEMS devices, such as micro channels and mixers for bio-MEMS[6,7], micro actuators and sensors[8,9] and terahertz components[10].

Various exposure techniques for the fabrication of ultra-thick SU8 microstructures with high aspect ratio, including UV, X-ray, e-beam and proton beam, have been demonstrated[11-13]. UV lithography is by far the most common exposure technique for patterning SU8 at low cost. However, the applications of SU8 have some severe problems. One of them is the large internal stress generated during the lithography process, and the other one is the poor adhesion on the substrates. Low post-exposure bake (PEB) temperature (55 °C) can significantly reduce the internal stress[14], but it takes much more processing time. Kyu-Youn Hwang et al[15] examined the adhesion behavior of SU8 microstructures on silicon (Si) and some other metal substrates, and the effect of the surface cleaning method on SU8 adhesion was first introduced. Another problem on the fabrication of high aspect ratio SU8 microstructures is that it is difficult to have nice straight sidewall fabricated by UV lithography compared with X-ray lithography. Also, the dimension changes from top layer to bottom layer by UV lithography, which influences the further application of SU8 microstructures significantly. Jun Zhang et al[16] developed a new diffraction-refraction-reflection model to investigate the effect of exposure dose on the replication fidelity and the profile of SU8 microstructures. K. D. Vora[17] gave a theoretical investigation of influence of parameters on the sidewall slopes and roughness of thick SU8 microstructure. However, these SU8 microstructures were both fabricated by X-ray lithography or UV lithography with a filter, and there is no exhaustive experimental analysis.

---

[*] This work has been supported by the National High Technology Research and Development Program (No.2015AA042603), and the Fundamental Research Funds for the Central Universities in China (Nos.106112014CDJZR160001 and 106112015CDJXY120002).

[**] E-mail: lidongling@cqu.edu.cn

The purpose of this work is focused on how to produce thick SU8 microstructures with high aspect ratios and good sidewall profile using broadband UV lithography. A comprehensive study of the fabrication of thick SU8 microstructures is investigated. The importance of adhesion on fabricating high aspect ratio SU8 microstructures is discussed, and the factors causing cracks and delamination are studied. Various processing parameters are optimized for obtaining the desired structures with accurate line width and good profile. Furthermore, the relationship between structure shape and fabrication process is analyzed. Finally, SU8 microstructures are used in the MEMS devices. The results shown in this paper are useful to optimize the UV lithography processes of SU8 photoresists and to improve the design and fabrication efficiency of some microstructures for MEMS applications.

10.16 cm <100> n-type Si wafers were used as substrates. The used photoresist and developer were SU8 2075 and SU8 developer supplied by Nanjing Baisiyou Company. For experimental purposes, a test mask was made with several common shapes, such as squares, hexagons, cylinders, line spaces and other partial closed field structures.

The wafers were first cleaned by immersing in piranha solution (H_2SO_4:H_2O_2=3:1, in volume) at 120 °C for 20 min followed by ten rinses of deionized (DI) water each lasting 1 min. To dehydrate the wafer, the acid cleaned wafer was then baked at 200 °C for 1 h in a convection oven. To obtain the film thickness of about 160 μm, SU8 2075 was spun onto Si wafer with the speed of 900 r/min. After the films were allowed to settle for a few minutes to eliminate bubbles and improve surface uniformity, they were heated with a controlled three-step soft bake, as shown in Fig.1. Before ramping to 65 °C, a 10 min stop is done at 50 °C to enhance the reflow of photoresist. And then, they were baked at 65 °C and 95 °C for 8 min and 45 min, respectively. The resulting films were then carefully cooled down to room temperature (RT) with a slow cooling rate. Care must be taken during heating and cooling ramps to avoid cracking and edge beading due to internal stress within the thin film. UV exposure of 288 W was applied to the films using a broadband mask aligner via hard contact mode for 35 s with the intensity of 23.3 mJ/cm^2 to induce the crosslinking of SU8 photoresist. A controlled two-step PEB was followed to selectively crosslink the exposed portions of the film. The accurate ramp rate and baked time were also shown in Fig.1. After the temperature was cooled down to 25 °C, the films were immersed into SU8 developer for 15 min, and then were rinsed into isopropanol (IPA) and DI water to remove the residual developer. Finally, the wafers were hard baked at 150 °C for 1 h. Additionally, the wafers must be kept horizontal in all the baking processes to prevent the non-uniformity caused by reflow of SU8.

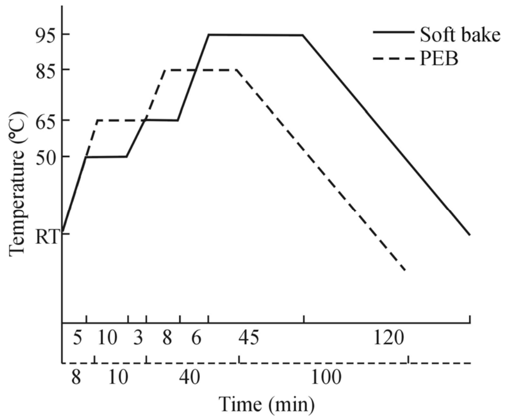


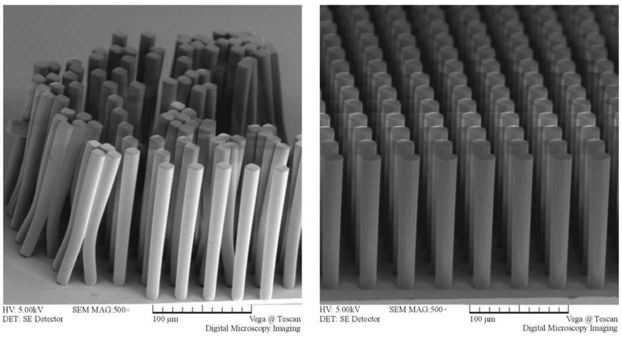
Fig.1 Experimental procedure for soft bake and PEB of the SU8

Because of the mismatch of the coefficient of thermal expansion (CTE) between SU8 films (50×10^{-6} /°C) and the Si substrates (2.6×10^{-6} /°C), large internal stress will produced during the SU8 fabrication process, which results in cracks and wrinkles in the fabricated SU8 microstructures. When working on a low adhesive surface, large stress will also distort or even totally peel the SU8 patterns off the substrates. Attention must be taken to avoid these adverse phenomena.

The strong adhesion of the SU8 photoresist onto a solid substrate is a pre-requisite for using as a mold or a structural element of the MEMS device. It is especially important in generating microstructures with high aspect ratio and small contact area for adhesion. Weak adhesion always causes collapse and delamination.

Adhesion of SU8 depends on the substrate material, but it can be improved in general by either modifying the surface of the substrate or reducing the interfacial stress during the process. The surface properties of Si wafer were modified by employing different cleaning techniques. The piranha solution cleaning technique was firstly employed. It can eliminate the adsorbed organics effectively. However, a thin chemical SiO_2 layer was grown, and the Si surface is full of Si-O bonds. In contrast, after immersing the substrate in the piranha solution, a further HF treatment was performed, which can etch the generated SiO_2 layer and make the Si surface hydrophobic terminated with Si-H. Fig.2(a) and (b) show the scanning electron microscope (SEM) micrographs of 20 μm micro cylinder arrays cleaned with piranha and piranha/HF, respectively. The results show that some of the microstructures cleaned with piranha are collapsed and peeled off at the edge of the array, but the microstructures cleaned with piranha/HF are stand well without any collapsed and peeled region. It is indicated that the hydrophobic treatment can enhance the adhesion of SU8 with Si substrate, and can be used to fabricate microstructures with higher aspect ratio. That is because HF treatment leads to the significant changes of the surface properties by reducing polar component stemming from

Si-OH groups and increasing the dispersion component, which suggests that the interfacial surface properties determine the adhesion properties between SU8 film and Si surface.

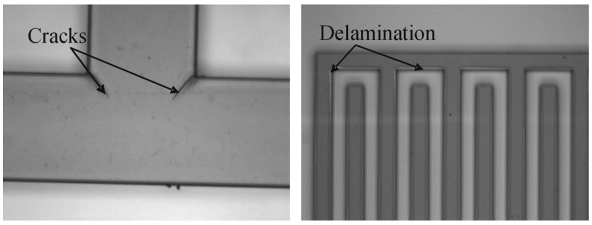


(a) (b)

Fig.2 SEM micrographs of SU8 micro cylinder arrays on Si substrate cleaned by (a) piranha and (b) piranha/HF

Cracks and delamination always occur after development, which are mainly caused by large internal stress induced by the shrinking of SU8. When the SU8 layer is not freestanding, the resulting shrinking will be translated into tensile stress. The shrinking going from developer to IPA occurs relatively fast, and the stress is at its highest in IPA. This is the main reason why cracks and delamination tend to occur at this point.

The cracks and delamination are significantly influenced by soft bake and PEB processes. The most important function of soft bake is to remove the solvent from SU8 layer. High solvent content will result in low strength of SU8 films, which results in bending of structures. Additionally, the low strength of SU8 is another factor of causing cracks[18]. In contrast, low solvent content will cause higher internal stress during heating and cooling procedures. Optimized soft bake was chosen to let the resist dry enough but still with some solvent for less internal stress and good mechanical properties. Experimental results show that 45 min soft bake at 95 °C is sufficient for 160 μm-thick SU8 films. For good uniformity and flatness, an additional bake at 55 °C for 10 min was used. The PEB process has the most important influence on the crosslinking and the shrinking of the SU8 film. On the one hand, the polymerization of SU8 can not complete at the PEB temperature less than 55 °C. On the other hand, higher PEB temperature results in higher internal stress, and makes the development hard. The recommended parameter of PEB process supplied by Microchem Company is 95 °C for 15 min. However, the cracks appear after development, especially at the corners of SU8 structures, as shown in Fig.3(a). Delamination of the SU8 patterns also appears as shown in Fig.3(b), even good cleaning is applied to improve the adhesion. When the PEB temperature is decreased to 85 °C for 40 min, the microstructures can be well fabricated without any cracks and deformation. The PEB time is also important for the crosslinking of the SU8 photoresist. As a chemical reaction, the amount of the crosslinking is determined by the PEB duration time at a given temperature. Unfortunately, the photo-generated acid will diffuse laterally during the PEB process, which can ruin the structures and make the development hard. Therefore, the shortest acceptable PEB time at a given temperature is preferred.

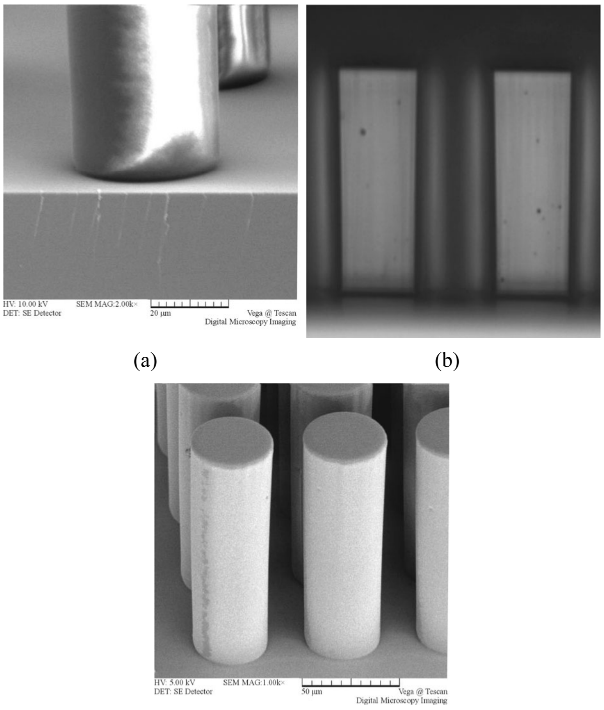


(a) (b)

Fig.3 Micrographs of (a) cracks and (b) delamination after development

The main aim of lithography is to accurately replicate the mask features into the resist. For using as a master mold for subsequent child mold generation, the profile characteristics, such as taper angle of the sidewall, are very important for the replication of microstructures. Controlling the taper angle of sidewall to a small but finite deviation from verticality is imperative for the use of SU8 microstructure as a mold for high aspect ratio replication.

In the experiments, “T-top” SU8 microstructures are obtained because of the use of wideband UV source, which results in line width difference and a slop profile. Jun Zhang found that prebake time plays the key role in the quality, both for resolution and aspect ratio. The exposure time and the PEB time have little effect on SU8 resolution[15]. In this article, some different results are obtained. Experimental results indicate that the exposure dose plays an important role in the line width and profile of SU8 microstructure. At a low dose (500 mJ/cm^2), there is not enough photo-initialized acid to polymerize the SU8, especially at the bottom. As a result, the undercuts easily appear, and the microstructures can be easily washed away by the developer, as shown in Fig.4(a). On the contrary, a high dose (1 100 mJ/cm^2) results in the expanded structures and slope sidewalls, as shown in Fig.4(b). The optimized exposure dose is 800 mJ/cm^2 with the SU8 thickness of 160 μm, and the obtained cylinders are shown in Fig.4(c). The SEM micrographs show the nearly vertical sidewall and slight undercut. The diameters of the micro cylinders are 45.6 μm and 44.8 μm at the top and bottom, respectively, and the taper angle is 89.86°.



(a) (b)

(c)

Fig.4 SEM micrographs of SU8 microstructures obtained with different exposure doses of (a) 500 mJ/cm^2, (b) 1 100 mJ/cm^2 and (c) 800 mJ/cm^2

Other problems related to profile are local non-uniformity and air bubbles of the SU8 layer, as illustrated in Fig.5. On a non-uniform surface, air will introduce another refractive surface, and light is bended to other regions which are not planned to be exposed, as line a shown in Fig.5. Lines b and d show the path of the light without refraction, and line c shows the refractive effect of an air bubble.

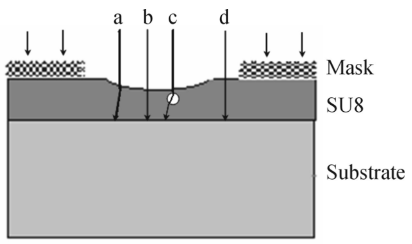
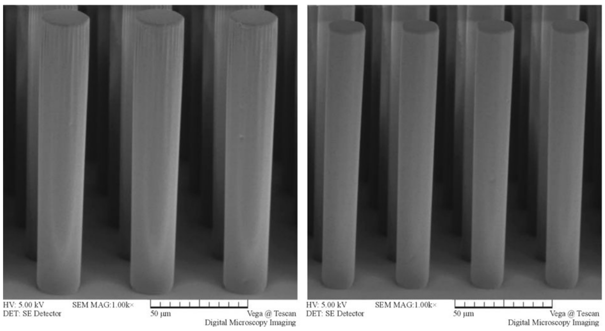


Fig. 5 Schematic diagram of the influence of non-uniformity and air bubble on the SU8 exposure

The diffraction, refraction and reflection significantly affect the SU8 profile, especially the taper angle, micro-channel width and roughness. To obtain uniform SU8 layers without air bubble, the wafers are kept horizontal in all the baking processes. After spin coating, the films are allowed to settle for a few minutes to eliminate bubbles and improve surface uniformity. The three-step soft bake process is another efficient way to improve surface uniformity and eliminate air bubbles. Fig.6 show the SU8 microstructures fabricated with different soft bake processes. The traditional two-step soft bake causes the non-uniformity of SU8 layers, and some craters or waves are obviously seen on the SU8 surface. Consequently, the fabricated micro pillars have rough profiles, especially on the top, as shown in Fig.6(a). The improved three-step soft bake allows the reflow of SU8 under T_g (glass transition temperature), and the surface of SU8 film is improved. The obtained micro pillars have good shape and smooth profile, as shown in Fig.6(b).

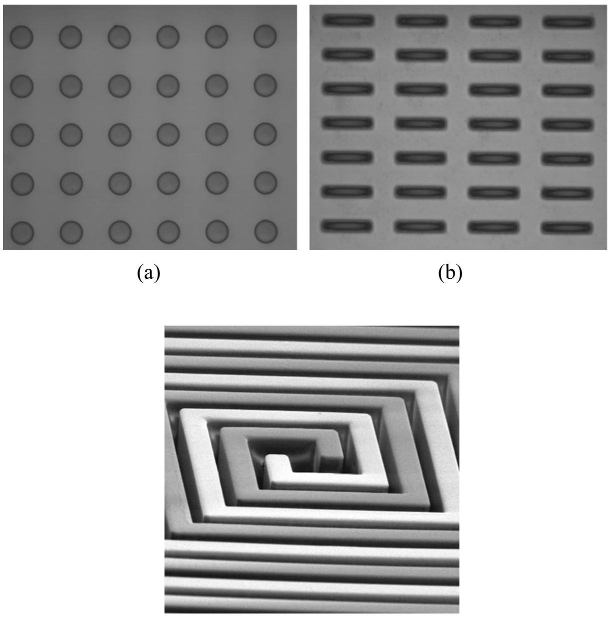


(a) (b)

Fig.6 SU8 micro pillars fabricated by traditional two-step soft bake and (b) three-step soft bake

For studying the effect of structure shape on the fabrication of SU8 microstructures, a test mask was made with several common shapes, such as independent squares, hexagons, cylinders, line spaces, interdigital, holes with different shapes, channels and some other partial-closed field structures. All of these structures are arranged in the array with different gaps. The results show that the cylinders need higher exposure dose and longer development time compared with other standard open field structures, due to partial exposure by diffracting light from nearby mask patterns. Because of the small contact area of the micro pillars, they need good adhesion and high mechanical properties to prevent being washed away by developer and water, so higher exposure doses are used, and the development time is controlled strictly. Fig.7(a) and (b) illustrate the micrographs of micro cylinders and micro holes under the same fabrication parameters. It is obviously that the micro cylinders have good shape, but the micro holes have some residual at the corner of holes.

Furthermore, the development time is varied for different structures as shown in Fig.8. The raised structures (micro square, cylinders and line spaces) need the shortest development time because of the large contact area with developer. The concave structure (holes and channels) and partial-closed field structures (spirality) need longer development time and hard to be developed clearly. The residual can be found at the edge of SU8 structures as illustrated in Fig7.(c). Experimental results also show that the SU8 structures are sensitive to the gaps. The bigger the gap, the easier the fabrication is. And there is a minimum space with the given thickness. When the space is smaller than this minimum value, it can not be separated even developed for a long time. For the microstructure with the thickness of 160 μm, the minimum gap is 15 μm. However, small pillar is hard to be fabricated because of the small contact area with the substrate. In our experiment, the minimum pillar obtained without collapse and deform is 15 μm with the thickness of 160 μm and the gap of 15 μm, and the aspect ratio is better than 10.



(a) (b)

(c)

Fig.7 The SEM micrographs of (a) micro cylinders with good shape, (b) micro holes with residual at the corner of holes and (c) partial-closed field structure with residual at the edge under the same fabrication parameters

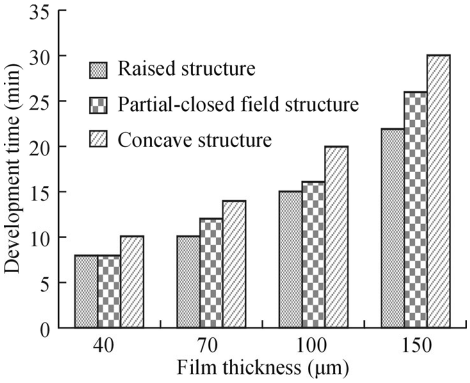
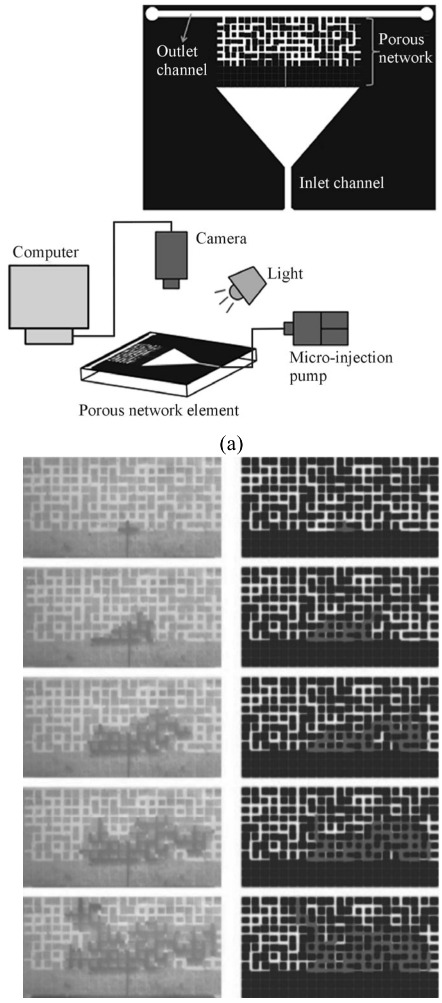


Fig.8 The development time of different structures with different thicknesses

SU8 has been widely used in our laboratory. For example, SU8 microstructures were used as etch mask in micro energy harvester, microstructures in supercapacitor and intermediate layers for wafer bonding. Here, detail results are given in the microfluidic device.

Replication rather than direct patterning is rapidly becoming the industry standard for the fabrication of polymeric microfluidics substrate because of low cost, ease of fabrication and desirable properties of polymers. For repeatedly using, the SU8 microstructures were firstly fabricated on Pyrex 7740 glass by standard UV lithography. Then, polydimethylsiloxane (PDMS) was pouring onto the substrate and solidified at 95 °C for more than 2 h. Finally, PDMS detached from the substrate and bonded with another glass substrate for further fluidic analysis. Two-phase transport characteristics were verified using this chip. The experimental apparatus are shown in Fig.9(a). The DI water was injected in the inlet channel by micro-injection pump, and the flow process of the liquid phase in the pore network channel was recorded by Sony DCR-PC110Ecamera. Fig.9(b) shows the experimental and numerical results of two-phase flow in bilayer pore network. The good conformity between experimental and numerical results shows that the fabricated SU8 micro-modes have little line width difference and smooth surface. Furthermore, this micromode can withstand more than 20 times repeated use.



(a)

(b)

Fig.9 (a) Schematic diagram of two-phase transport experimental apparatus using SU8 as the molds; (b) Experimental (left) and numerical (right) results of two-phase flow in bilayer porous network

Using conventional UV lithography, SU8 microstructures with good adhesion and vertical side wall were fabricated. The results show that the optimized process parameters and the proper structure design make the fabrication of SU8 microstructures with high aspect ratio feasible. The adhesion of SU8 films on Si substrate is influenced by the existence of Si-OH on the surface, and can be improved by the HF treatment. Cracks and delamination are caused by large internal stress during fabrication process, and are significantly influenced by soft bake and PEB processes. Exposure plays an important role in the line width and profile of SU8 structures. Low exposure dose results in undercut, and high exposure dose causes expanded structures and slope sidewalls. By applying a three-step soft bake and a PEB at low temperature (85 °C) with long time (40 min), the cracks and delamination are eliminated. The optimized exposure dose is 800 mJ/cm^2 with the SU8 thickness of 160 μm. Dense SU8 micro pillars are achieved with the aspect ratio of 10. The fabricated SU8 microstructures show a strong adhesion to Si substrates without any collapse, and have good profile with the taper angle of 89.86°. Some possible applications of SU8 in microfluidic systems and MEMS devices are also illustrated.

References

[1] Daniel Rodriguez Ponce, Karen Lozano, Thomas Eubanks, Ahmad Harb, Domingo Ferrer and Yuankun Lin, Journal of Polymer Science: Part B: Polymer Physics **48**, 47 (2010).

[2] Sofia Rahiminejad, Elena Pucci, Sjoerd Haasl and Peter Enoksson, International Journal of Microwave and Wireless Technologies **6**, 459 (2014).

[3] Matthew Holmes, Jared Keeley, Katherine Hurd, Holger Schmidt and Aaron Hawkins, Journal of Micromechanics and Microengineering **20**, 115008 (2010).

[4] Hou Shuang-yue, Xiong Ying, Chen Shan, Xiong Peng-hui, Chen Xiangyu, Zhang Xiaobo, Tian Yangchao and Liu Gang, Optics Express **10**, 0088 (2014).

[5] Srinivasa Reddy Kuppireddi, Sayanu Pamidighantam and Oddvar Sorasen, International Journal of Materials Science and Engineering **1**, 79 (2013).

[6] George Kumi, Ciceron O Yanez, Kevin D. Belfieldbc and John T. Fourkas, Lab on a Chip **10**, 1057(2010).

[7] Tijjani Adam and U. Hashim, Microsyst Technologies **22**, 433 (2016).

[8] Leema Rose Viannie, G. R. Jayanth, V. Radhakrishna and K. Rajanna, Journal of Microelectromechanical Systems **25**, 125(2016)

[9] Shang Yana, Ni Qingyan, Ding Ding, Chen Na and Wang Tingyun, Optics Express **11**, 0061 (2015).

[10] Yingtao Tian, Xiaobang Shang, Yi Wang and Michael J. Lancaster, Journal of Micro/Nanolithography, MEMS, and MOEMS **14**, 044507 (2015).

[11] Charles Becnel, Yohannes Desta and Kevin Kelly, Journal of Micromechanics and Microengineering **15**, 1242 (2005).

[12] A del Campo and C Greiner, Journal of Micromechanics and Microengineering **17**, R81 (2007).

[13] J. Liu, B. Cai, J. Zhu, G. Ding, X. Zhao, C. Yang and D. Chen, Microsystem Technologies **10**, 265 (2004).

[14] Bo Li, Miao Liu and Quanfang Chen, Journal of Micro/Nanolithography, MEMS, and MOEMS **4**, 043008 (2005).

[15] Kyu-Youn Hwang, Chin-Sung Park, Joon-Ho Kim, Kahp-Yang Suh, Eun-Chul Cho and Nam Huh, Journal of Micromechanics and Microengineering **20**, 117001 (2010).

[16] Jun Zhang, Mary B. Chan-Park and Samuel R. Conner, Lab on a Chip **4**, 646 (2004).

[17] K. D. Vora, B. Y. Shew, B. Lochel, E. C. Harvey, J. P. Hayes and A. G. Peele, Microsystem Technologies **14**, 1701 (2008).

[18] Kristof Wouters and Robert Puers, Journal of Micromechanics and Microengineering **20**, 095013 (2010).