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Polymerization optimization of SU-8 photoresist and its applications in microfluidic systems and MEMS

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Abstract

In this paper, SU-8 EPON-based photoresist (PR) polymerization optimization and its possible microfluidic and MEMS applications are reported. First, the optimization results of SU-8 under UV lithography are reported. The parameters which could have an influence on the lithography quality were chosen and optimized by a three-level, L9 orthogonal array of the Taguchi method. By optimization, the optimal parameter range and the weighted per cent of a parameter on the final results were determined. For SU-8-5 and SU-8-50, many microstructures with thicknesses of more than 100 and 500 μm and aspect ratios of more than 20 and 50 were obtained with high resolution. The optimization results show that the prebake time plays the key role in the quality, which is different from the previously published results. With the optimization results obtained, some possible applications of SU-8 were developed and demonstrated. These applications included using SU-8 as a structural material for a microfluidic system, as a micromold for electroplating, as a master for plastic hot-embossing, and even as a mask for some wet-etching processes.

(Some figures in this article are in colour only in the electronic version; see www.iop.org)

1. Introduction

UV-LIGA is an important technology for the realization of some very thick microstructures with high aspect ratios for MEMS applications [1–3]. The core difficulty of this technology is the use of UV or near-UV lithography to form very thick micromolds for electroplating. With commonly-used positive photoresists (PRs), only tens of micrometers can be achieved. This has restrained the further application of UV-LIGA technology. EPON SU-8 series negative PR has been proven the most important substituent for this application since thick molds (>1 mm) with high aspect ratios (>50) can be achieved [4–13]. However, in practical use, SU-8 has been shown to be sensitive to process parameter variation. The parameter values described in the literature vary in a wide range and no further studies have yet been performed to decide which parameters are most crucial to achieve good SU-8 microstructures.

The purpose of this work is focused on how to produce microstructures with high aspect ratios and resolution and how to use them in microfluidic systems and microelectromechanical systems (MEMS). First, a three-level L9 orthogonal array in the Taguchi method is used to optimize the processing parameters. Then, the potential applications of SU-8 are demonstrated. All these show that by optimizing the parameters, SU-8 is a potential material for use in some MEMS or microfluidic applications in the future.

2. Taguchi optimization of SU-8 polymerization

2.1. SU-8 EPON PR and UV-polymerization

SU-8 (Microchem Corporation) is a negative, epoxy-type, near-UV PR based on the EPON SU-8 resin. It can be obtained by dissolving the EPON resin SU-8 in the organic solvent GBL (gamma-butyloractone). The quantity of the solvent determines the viscosity and hence the possible thickness range of the resist. Triaryl sulfonium salt is added (10 wt% of EPON

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Table 1. Parameter settings of the SU-8 photoresist for the Taguchi optimization.

Thickness (μm)	Level	Prebake (min)	Exposure (s)	Postbake (PEB) (min)	Develop (min)
SU-8-5					
40	1	12.5	28	20	5
	2	15	29	25	6
	3	20	30	30	7
30	1	10	17.5	15	4
	2	12.5	18.5	20	5
	3	15	19.5	25	6
13	1	8	10	15	3.5
	2	10	10.5	20	4
	3	15	11	22	5
SU-8-50					
275	1	40	100	40	13
	2	50	105	50	16
	3	60	110	60	18
150	1	35	50	35	10
	2	45	52.5	45	12.5
	3	55	55	55	15
95	1	25	35	25	8
	2	35	40	35	10
	3	45	45	45	12

Table 2. The optimal setting of the resolution for the SU-8-5 photoresist. In tables 2–5 ^{7} denotes representative significant factors.

	Thickness					
	40 μm		30 μm		13 μm	
Parameters	Raw data	S/N ratio	Raw data	S/N ratio	Raw data	S/N ratio
Prebake	20 min^7	20 min^7	12.5 min^7	12.5 min^7	10 min^7	8 min^7
Expose	28 s^7	30 s^7	18.5 s^7	18.5 s^7	10 s^7	10 s^7
Postbake	30 min	30 min	25 min^7	25 min	20 min	20 min
Develop	7 min^7	7 min^7	6 min^7	6 min^7	5 min^7	5 min
Optimum	5.341	11.698	4.933	10.148	4.70	11.14
99% confidence interval	±0.463	±0.485	±0.619	±4.025	±0.506	±0.841
Confirmation results	5.721	11.711	5.354	14.012	5.403	11.523

SU-8) and mixed with the resin as a photoinitiator [8]. The two series of SU-8-5 and SU-8-50 were chosen in this paper because they allow a wide coverage of structural thicknesses (from 10 to 350 μm for a single-layer coating and more than 1 mm for multi-layer coating). This wide coverage of thickness could satisfy most MEMS applications. Moreover, studies on these two series have not yet been reported in the literature.

All experiments were processed on 4 inch Si wafers with a native oxide layer. First, the resist was spin coated at different rotation speeds and baked at 95 °C on a hot plate. Then, the resist was exposed to UV light with an intensity of 10 mW cm^{-2}. Next, propylene glycol methyl ether acetone (PGMEA) was used as the developer. An ultrasonic agitator was used to increase the developing speed. Finally, the quality of obtained PR structures was assessed on their resolution and aspect ratio. The thickness was determined by a profiler. The thickness readings for the resolution were chosen from five different locations of the wafer with the three highest readings used for optimization. The aspect ratios were obtained by the combination of scanning electron microscopy (SEM) photographs and profiler results.

A schematic diagram of the UV-LIGA technology is shown in figure 1. The PR was spin coated onto the substrate. Under UV lithography, the PR is polymerized and selectively forms the microstructures. Since the substrate is electrically conductive, electroplating can proceed only in the exposed window areas. After plating, we can remove the PR micromolds to obtain the metallic structures.

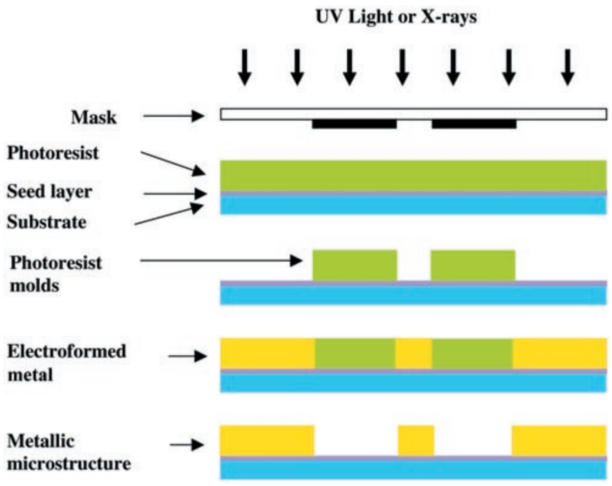


Figure 1. Schematic diagram of the UV-LIGA technology.

Table 3. The optimal settings of the aspect ratio for the SU-8-5 photoresist.

Parameters	40 μm Raw data	40 μm S/N ratio	30 μm Raw data	30 μm S/N ratio	13 μm Raw data	13 μm S/N ratio
Prebake	20 min^{7}	20 min^{7}	15 min^{7}	15 min^{7}	15 min^{7}	15 min^{7}
Expose	30 s^{7}	30 s^{7}	18.5 s^{7}	18.5 s^{7}	10.5 s^{7}	10.5 s^{7}
Postbake	30 min^{7}	30 min^{7}	20 min^{7}	20 min	15 min^{7}	15 min^{7}
Develop	7 min^{7}	7 min	6 min^{7}	6 min^{7}	5 min^{7}	5 min
Optimum	7.333	−32.12	21.67	−36.29	50	−36.97
99% confidence interval	±6.64	6.922	±5.96	±4.86	±6.19	±2.76
Confirmation results	5.012	−28.21	16.31	−33.11	48.01	−32.56

Table 4. The optimal settings of the resolution for the SU-8-50 photoresist.

Parameters	275 μm Raw data	275 μm S/N ratio	150 μm Raw data	150 μm S/N ratio	95 μm Raw data	95 μm S/N ratio
Prebake	40 min^{7}	40 min^{7}	45 min	45 min^{7}	25 min^{7}	25 min^{7}
Expose	100 s	100 s	52.5 s	52.5 s	40 s^{7}	40 s^{7}
Postbake	40 min	40 min^{7}	55 min^{7}	55 min^{7}	25 min^{7}	25 min^{7}
Develop	18 min^{7}	18 min^{7}	15 min^{7}	15 min^{7}	10 min^{7}	8 min
Optimum	4.904	10.993	3.833	8.344	5.133	9.948
99% confidence interval	±0.408	±0.903	±0.465	±3.400	±0.607	±3.798
Confirmation results	4.9	11.02	4.8	11.431	5.0	11.872

2.2. Taguchi optimization methodology [14]

Some preliminary experiments had shown that many parameters could affect the quality of SU-8 polymerization. The Taguchi statistical method is an effective method to obtain the optimum data range of the process parameters. The orthogonal arrays selected for the three levels were L9 orthogonal arrays. Analysis of variance (ANOVA) was used to interpret the experimental results and decide which level of the factor should be used. An F-test was also performed to find the significance level of the factors. The significant factors with their respective levels were identified and set to the optimum values. Once the optimum response was predicted, the confidence interval was then calculated. The final step of the Taguchi method was to conduct confirmation tests and verify the results obtained with the optimum parameter settings.

The experiments were conducted as described by an orthogonal array; the various parameter settings are shown in table 1. The four parameters selected to improve the characteristics are the prebake time, exposure time, postbake time and development time. A three-level L9 orthogonal array is used to study the effects of the four control factors, including the prebake time, the exposure time to 10 mW cm^{-2} near-UV, the post-exposure time, the baking time and the developing time on three different thicknesses of the PR films achieved by the three different spin speeds. The evaluation standard was to gain height characteristics, such as high resolution and aspect ratio, which can be appraised with SEM and the profiler.

2.3. Optimization of the SU-8 negative PR

The parameter settings for the three different thicknesses (40, 30 and 13 μm for the SU-8-5 and 275, 150 and 95 μm for the SU-8-50) are shown in tables 2–5. The quality characteristic change of a product under investigation, corresponding to a factor introduced in the experimental design, is termed as the signal of the desired effect. When an experiment is conducted, numerous external factors, not designed, will be involved in the experiment and affect the outcome. These external factors are called the noise factors and their influence on the outcome of the quality characteristics under test is termed as noise. Thus, the ratio of the signal to the noise (S/N) demonstrates the sensitivity of the quality characteristics, being investigated in a controlled manner, to some other external factors not under control. The S/N ratio is also applied to analyze the raw data. The aim of the optimization experiment is always to determine the highest possible S/N ratio for the result since a high S/N ratio implies that the signal is much higher than the random effect of the noise factor. In this experiment, the S/N ratio of resolution is measured with the 'higher the better' characteristic while the S/N ratio of the aspect ratio is measured with the 'lower the better' characteristic. (In tables 2–5, * denotes representative significant factors, or RSFs.)

3. Analysis and discussion

Through the experimental results shown in figures 2–5, it can be seen that the influence of each parameter on the quality of the microstructures is remarkable. However, the per cent contribution of these factors to the final result varies over a very large range.

3.1. SU-8-5

The per cent contributions of the four selected parameters to the resolution and aspect ratio are shown in figure 6(a) and (b), respectively. From figure 6(a), the contribution ratios of the four factors to the final result are different, varying from

Table 5. The optimal settings of the aspect ratio for the SU-8-50 photoresist.

	275 μm		150 μm		95 μm	
Parameters	Raw data	S/N ratio	Raw data	S/N ratio	Raw data	S/N ratio
Prebake	40 min[7]	40 min[7]	55 min[7]	45 min[7]	35 min[7]	35 min[7]
Expose	105 s[7]	105 s	50 s	50 s	45 s[7]	45 s[7]
Postbake	50 min	50 min[7]	55 min[7]	52.5 min[7]	45 min[7]	45 min[7]
Develop	18 min[7]	16 min[7]	15 min[7]	15 min[7]	10 min[7]	10 min
Optimum	1.7×10^{15}	-19.92	8.667	-27.939	2.333	-28.132
99% confidence interval	± 4.526	± 15.261	5.241	4.464	6.193	6.944
Confirmation results	0.01	-12.541	3.01	-23.976	0.1	-23.475

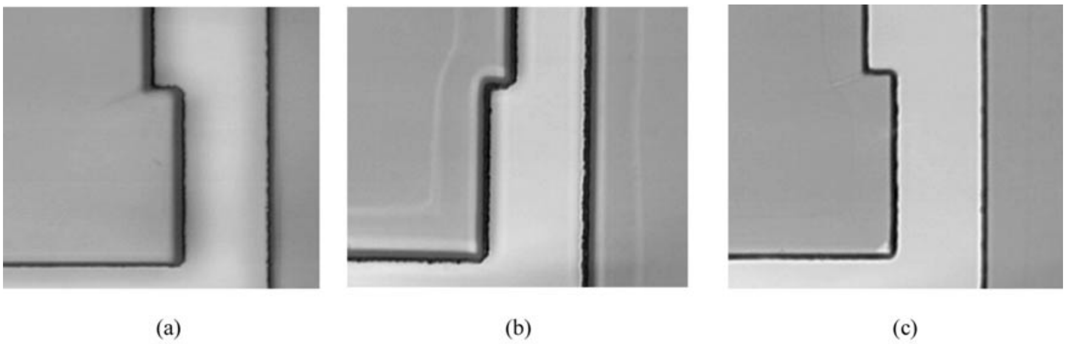


Figure 2. Confirmation result of the SU-8-5 PR for resolution: (a) 40 μm, (b) 30 μm and (c) 13 μm.

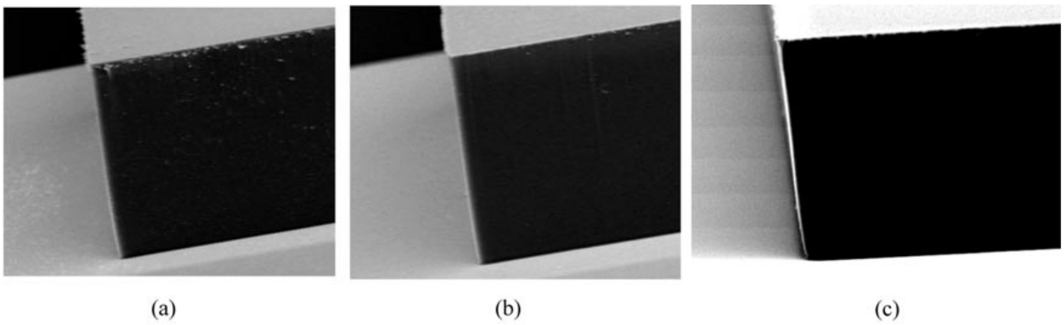


Figure 3. Confirmation result of the SU-8-5 PR for aspect ratio: (a) 40 μm, (b) 30 μm and (c) 13 μm.

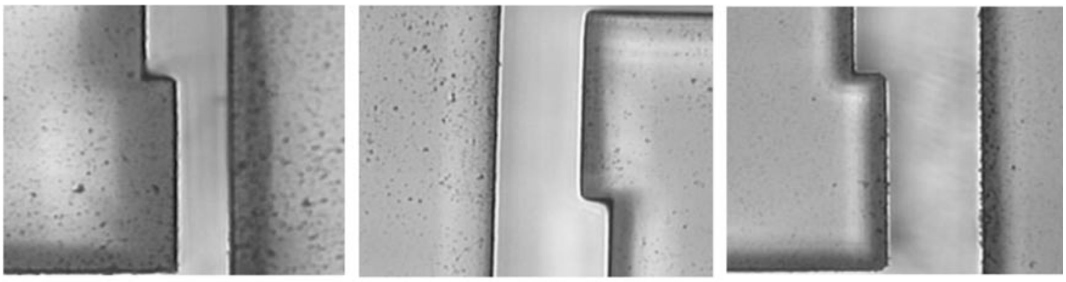
prebake, 42.37%, to postbake 4.6%. The prebake time is the most important factor to the resolution. From figure 6(b), the prebake time (47.02%) was also the key factor. Therefore, for SU-8-5, good resolution and a high aspect ratio can be obtained at the same time by carefully controlling the prebake time. The experimental results also show that as the PR structure gets thicker, the influence of the developing time increases. The reason is that more SU-8-5 needs to be developed, so we require a more precise control of the exposure time. In addition, as the structure gets thicker, it becomes more difficult for the developing solution to reach the bottom of the PR and this will greatly reduce the efficiency of the developing solution.

3.2. SU-8-50

The optimization results of SU-8-50 are shown in figure 7(a) and (b). Figure 7(a) shows that the prebake time was a highly significant factor (40.70%). The developing time was the second most important factor (22.06%). The trend that the influence of the developing time will increase with increasing thickness can be seen. The exposure time and the postbake time have little effect on SU-8-50 resolution.

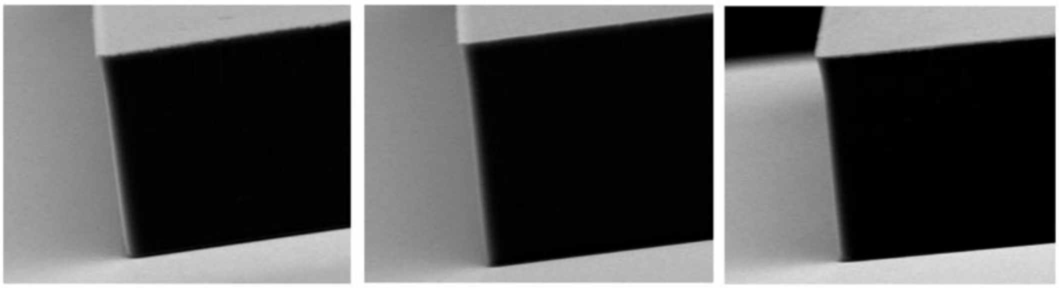
From figure 6(b), the prebake time (27.52%), developing time (24.65%) and exposure time (24.17%) have almost identical influences on achieving a higher aspect ratio. Once again, there also exists a trend that the developing time will become more significant in the thicker photoresists. From the optimized settings for the resolution and aspect ratio of SU-8-50, we can find that layers with thicknesses of 275 and 150 μm show almost similar settings; but for a thickness of 95 μm, some differences occur. It is found that the prebake time and the developing time are highly significant. However, it is noted, from the aspect ratio optimization, that the exposure time becomes more significant. This is because the aspect ratios of the photoresist structures are more sensitive to the exposure dosage as the difference between the three-level setting of the exposure time is only 1–2 s. This effect is also seen in the SU-8-5 aspect ratio optimization.

For the four parameters we had selected, the most important factor is the prebake time. This conclusion is



(a) (b) (c)

Figure 4. Confirmation result of the SU-8-50 PR for resolution: (a) 275 μm, (b) 150 μm and (c) 95 μm.



(a) (b) (c)

Figure 5. Confirmation result of the SU-8-50 PR for aspect ratio: (a) 275 μm, (b) 150 μm and (c) 95 μm.

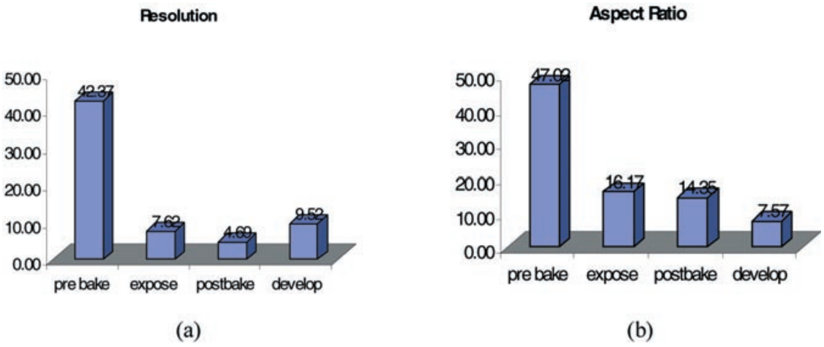


Figure 6. Per cent contributions of the different parameters on the final results of SU-8-5.

different from the previously published optimization results, which demonstrated that the exposure time was the key factor [15]. Since the prebake time for SU-8 is to evaporate the solvent (80 wt% or so), we think that a good resolution and aspect ratio only corresponds to a fixed solvent content. Similar results have also been found for other PR lithography [1], so our results seem more feasible and reasonable.

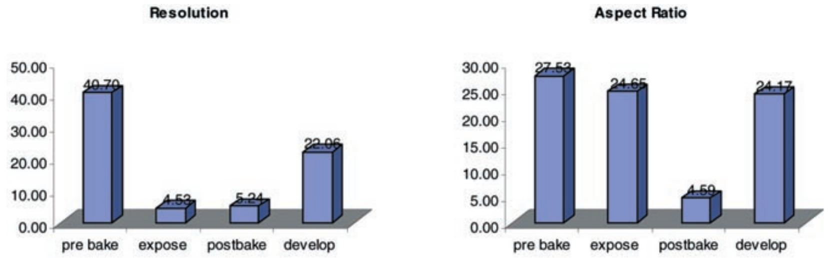
The exposure time is the most important factor and this can be shown in traditional PR applications. For very thin SU-8 films, the result also seems different, especially when the PR thickness increases. When the thickness is several hundreds of micrometers, the novel difference is that the exposure time does not cause the expected changes of the aspect ratio and resolution when the other parameters are fixed. We can, however, observe the changes induced by the prebake time variation.

4. Applications of SU-8 in microfluidic systems and MEMS

SU-8 has been widely used in our laboratory. The possible applications of SU-8 in microfluidic systems and MEMS can be demonstrated as below.

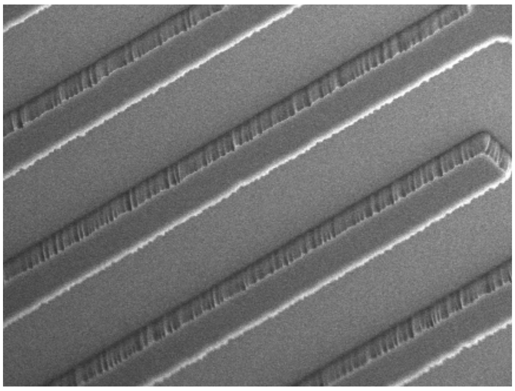
4.1. As micromolds for electroplating

At present the most difficult problem in using SU-8 in electroplating is how to remove the SU-8 micromolds after the electroplating is finished so we tried to solve the problem as follows. First, a positive PR, AZ 9260, was spin coated onto a silicon wafer (about 2 μm) and baked for 2 h. Then, a SU-8 negative PR was coated and developed with the optimized values. After 20 min postbake at 95 °C, the exposed films were dipped into the SU-8 developer. By choosing the proper developing time, we can wash away the unexposed SU-8 layer and leave the positive PR layer undamaged. Then, the positive

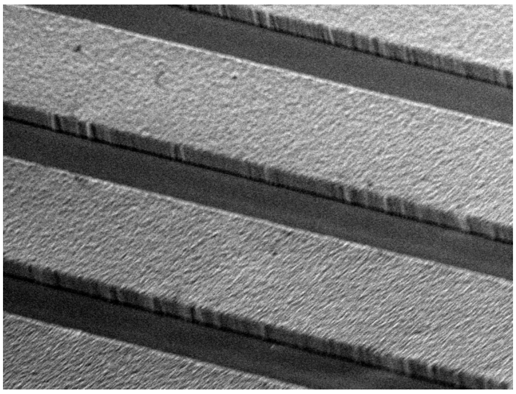


(a) (b)

Figure 7. Per cent contributions of the different parameters on the final results of SU-8-50.



(a)



(b)

Figure 8. Electroplating micro heat pipe using SU-8 as the molds: (a) SU-8 molds and (b) Cu electroplating microstructure.

PR layer was etched away by drifting in an acetone solution for several seconds. The undercut caused by the positive PR attacked by the acetone is very small and the error is tolerable for the next electroplating process. No hardbake was used after the lithography. After electroplating, the molds can be removed easily by acetone. Using this method, we fabricated a Cu and Ni microheat pipe. The SEM photograph is shown in figure 8.

4.2. As the master for hot-embossing

Hot-embossing is an effective method to obtain some low-cost microstructures (with a thickness of less than 100 μm) for some biochip applications. Patterned SU-8 (as master) and polycarbonate (with a glass transition temperature T_g of 145 °C) or PMMA (with T_g of 105 °C) substrate are fixed into two plates. The two plates were also used as the hotplates for heating and cooling applications. A force, 20 000 N in our case, was applied to the 'mates' by a force frame. After the hotplates were cooled down, the embossing tool was mechanically driven apart from the substrate, thus obtaining some microchannels for batch bioMEMS applications. A SEM photograph is shown in figure 9. However, the problem of how to remove the SU-8 molds from the substrate effectively still exists.

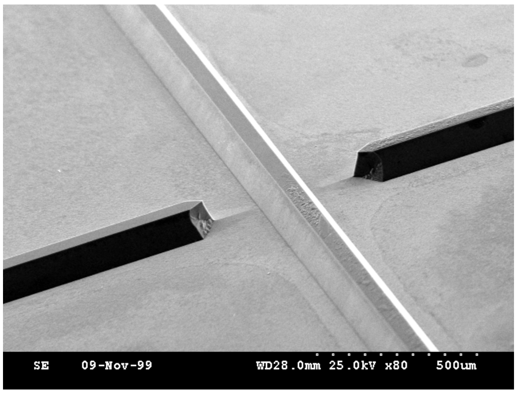


Figure 9. Microstructure obtained by polycarbonate hot-embossing using SU-8 as the master.

4.3. As structural material to construct the fully SU-8 polymer structure

Using positive AZ9620 as the sacrificial layer, a fully SU-8 polymer structure can be constructed in which the microchannels or microcavities are embedded in the SU-8 structure. A SEM photograph is shown in figure 10. The thickness of the microchannel can be up to 20 μm, depending upon the thickness of the AZ9620. In order to construct the fully SU-8 structures, two or more photolithography processes are necessary. The whole process is as follows. The first SU-8 layer was coated and patterned. Next, without a developing step, some thin films, such as metal films, parylene films, etc, were deposited on the SU-8 surface and used as an insulation layer. The insulation layer was patterned, leaving the alignment marks for the next step. Then, the second SU-8 layer was spin coated and patterned. Finally, the wafers were dipped into the developer agitated, with ultrasound, and the final microstructures can be obtained. The introduction of the insulation layer can diminish the interference effectively induced by the multi-exposure process.

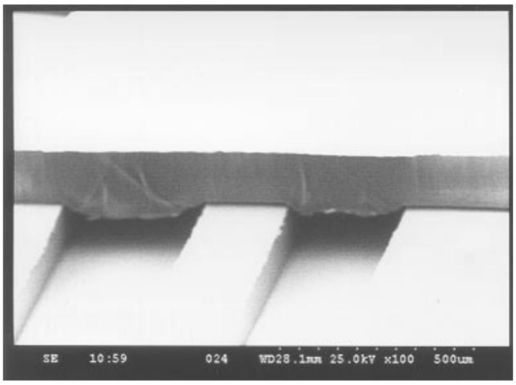


Figure 10. Embedded microchannel using AZ9620 as the sacrificial layer.

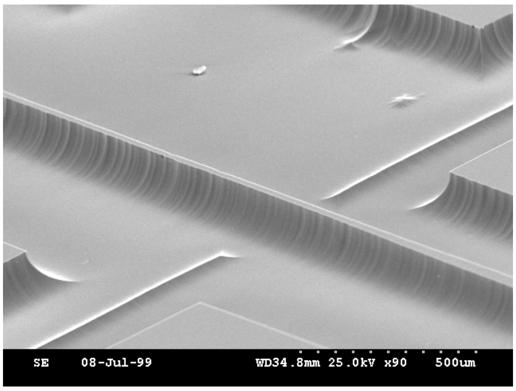


Figure 11. Boro-glass wet etching with SU-8 as the mask material.

4.4. As a mask layer for some wet etching processes

SU-8 has very good adhesion with the substrate after postbake. We tried to use SU-8 as the mask material for glass etching. It is well known that the mask choice in glass etching process is very important. The commonly used Cr/Au/PR composite layer cannot resist the HF-based etchant for a long time because of pin-holes and poor adhesion. Using SU-8 as the mask, we can obtain an etching depth of more than 100 μm easily and the SU-8 mask was not significantly damaged. A SEM photograph is shown in figure 11. After etching, we can remove the SU-8 mask by dipping it in hot pure H_2SO_4 for a long time or heating it with O_2 in a furnace, at up to 300 °C. The undercut, similar to Cr/Au/PR, is also a problem for SU-8 as the mask. This is due to the isotropic property of the glass; but the pits on the surface caused by the pin-holes in the mask can be dramatically decreased. The rough sidewall surface also can be improved by annealing the etched structures in an oven at a temperature below 350 °C.

5. Conclusion

The Taguchi optimization technique has been applied to two commercially available SU-8, negative photoresist series, SU-8-5 and SU-8-50. From the experiment results, it is found that all four control factors, prebake time, exposure time, postbake time and developing time, are significant in order to achieve high resolution and aspect ratios. For the four factors we had selected and optimized, the prebake time is the most significant one for all the six photoresist layers, of different thicknesses. However, the influence of the developing time increases with increasing thickness. Some possible applications of SU-8 in microfluidic systems and MEMS were also illustrated.

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