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Process development for the formation of post-bonding biorecognition layers in microfluidic biosensors

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Abstract - **Formation of the biorecognition layers within microfluidic sensor channels must be done after the completion of the channel structure since these layers cannot withstand the wafer bonding temperature. We propose a new post-bonding immobilization process to prepare the enzyme layers within microfluidic channels of electrochemical biosensors. An array of Pt vertical electrodes is electroplated using a SU-8 mold. The cured SU-8 is then removed by plasma etching to expose the Pt electrode and to define the fluidic channel cavity simultaneously. An array of enzyme posts is formed on the Pt surface by either electropolymerizing or photopolymerizing enzyme precursor solutions injected into the channel. Very little report is available about the reactive ion etching (RIE) of SU-8 that is the most critical step in this process. A systematic investigation on the RIE of SU-8 is conducted to obtain the maximum etch rate (1.2 µm/min.) based on different combinations of parameters of CF_4 and O_2 gas flow, RF power and time.**

I. INTRODUCTION

Bioanalytical microfluidic sensors have a great potential for the determination of multiple analytes during on-line monitoring. One of the bottleneck technologies is the preparation of biorecognition layers (e.g. enzyme, antibody, receptors, etc.) on selective areas within microfluidic channels. It becomes very challenging especially when these layers are to be prepared after the completion of fluidic channel structure by wafer bonding processes.

We are developing a new fabrication process as in Figure 1 to address this issue. The SU-8 is utilized as the mold to electroplate Pt vertical electrodes. These electrodes serve as the active sensing electrodes on which electropolymerized enzyme posts (polyaniline) or photopolymerized enzyme posts (polyvinyl alcohol) [1] will be immobilized. Unpolymerized solutions are flushed away from the channel. The vertical electrode [2] is preferable over the planar thin film electrode, especially for the photopolymerization as in Figure 1 (f), for a broader range of volume/area ratio (enzyme column volume vs. electrode surface) to manipulate the sensitivity and response time.

The most critical step in this process is the selective removal of cured SU-8 after use. Once polymerized, the SU-8 photoresist is difficult to remove since it is supposed to form a permanent part of devices in most cases. Reported reactive ion etching (RIE) methods have used an O_2-CF_4 plasma in ratios of 50%:50% and 75%:25%, respectively [3]. Oxygen etch chemistries with ~5 vol. % SF_6 also appeared to give enhanced etch rates [4]. We conducted a systematic investigation to obtain the highest etch rate using different combinations of process parameters of CF_4 gas flow, O_2 gas flow, RF power and time. We present this new post-bonding fabrication process

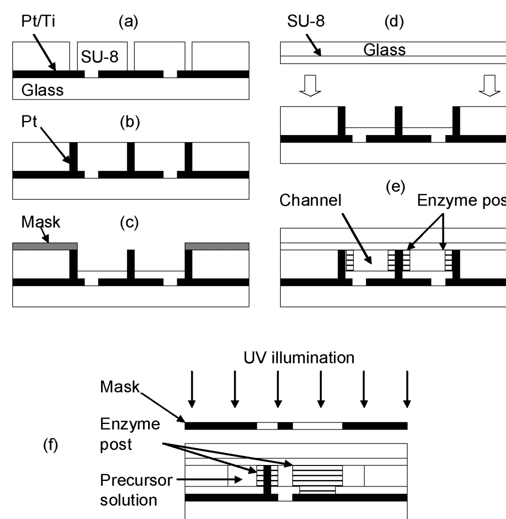

Fig. 1. Prospective process sequence. (a) Pt/Ti deposition and patterning, SU-8 patterning. (b) Pt vertical electrode electroplating. (c) Ti deposition and patterning, SU-8 reactive ion etching, Ti wet etching. (d) Glass/SU-8 cover layer bonding. (e) Enzyme post by electro- or photopolymerization of injected precursor solutions. (f) Comparison between the vertical electrode and planar electrode with photopolymerized enzyme columns.

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of microfluidic biosensors focusing on the reactive ion etching for selective removal of SU-8 and the thermal bonding of SU-8 wafers.

II. EXPERIMENTALS

A. SU-8 reactive ion etching (RIE)

The reactive ion etch (RIE) process was optimized using a 2^3 full factorial design and an analysis of variance (Table 1). The measured etch rate was the only response variable, and all statistical analyses were analyzed with Minitab statistical software at the 95% confidence level (α = 0.05) [5-7]. The SU-8 was spin coated on four inch Si wafers. The etch process was carried out on fully polymerized SU-8, which underwent soft bake steps at 65ºC and 95Cº, exposed to broadband UV light at 300mJ/cm^2, post exposure baked at 50º, developed for 6 minutes, and hard baked at 200ºC. All etch experiments were carried out using a Plasma Etch PE-200 reactive ion etch system. The changes in SU-8 surface roughness were measured with a KLA Tencor Alpha Step 200, and goniometry determined if the etched SU-8 surface was hydrophilic or hydrophobic.

Polyimide tape served as the temporary etch mask for SU-8 patterning. Linear trenches ~25µm deep were patterned in ~50µm thick SU-8 films. The width of the trenches varied from 250 to 700µm. Dry resist film was also used as a dry etch mask on some trials not related to the factorial design.

Factor	High	Low
RF Power, W	280	200
CF_4 flow, cc	10	5
O_2 flow, cc	95	90

Table 1. 2^3 factorial design for reactive ion etch of SU-8 2050. Process time was kept constant at 90 seconds.

B. SU-8 wafer bonding

Fully polymerized SU-8 was bonded to partially polymerized SU-8 [8-10]. The wafers were UV exposed for 50, 70 and 100mJ/cm^2 followed by a short post exposure bake step at 50ºC. The develop and hardbake steps were excluded. Bonding was done with a Dynatex DXB wafer bonder at 117ºC and 25 in-Hg pressure for 45, 60 and 70 minutes. Wafer bond strengths were determined using a Romulus Universal Materials Tester.

III. RESULTS & DISCUSSION

A. SU-8 reactive ion etching (RIE)

The 2^3 factorial design showed that RF power, CF_4 gas flow, and the RF-CF_4 interaction had a significant effect on the SU-8 etch rate (Figure 2). These terms had p-values < α, and their effects were statistically significant. The highest etch rate was ~1.2 µm/min. at 90 sccm O_2, 10 sccm CF_4, and 280W (Table 2). The CF_4 gas flows of 10 sccm (~10 vol. %) along with higher RF power may have created sufficient F that interacted with the SU-8 backbone and created reactive sites for O to form volatile compounds [11] (RF-CF_4 interaction in Figure 3). Etch trials with a CF_4 gas flow >>10 vol. % only increased the etch rate to ~1.5 µm/min., possibly a result of the formation of CF_2 (teflon) during processing. The baseline process for etching patterns or trenches on SU-8 was therefore set at 90sccm O_2, 10sccm CF_4, and 280W (Figure 4).

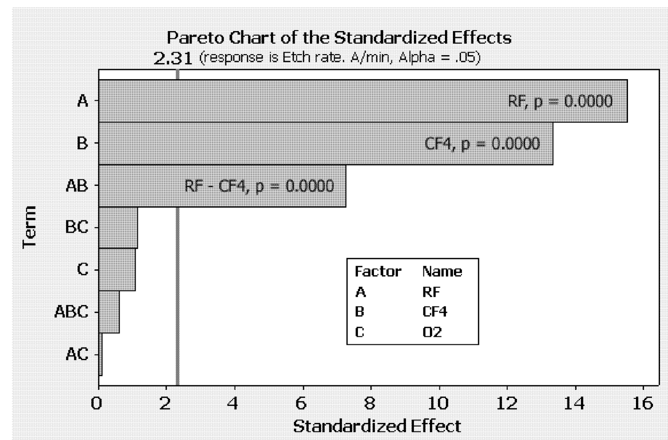

Fig. 2. Effects Pareto showing the influence of RF power, CF_4, and the RF-CF_4 interaction (p-value < α = 0.05).

RF Power, W	CF_4, sccm	O_2, sccm	Avg. Etch Rate, µm/min (+/- stdev.)
200	5	90	0.42 +/- 0.18
200	5	95	0.43 +/- 0.16
200	10	90	0.64 +/- 0.26
200	10	95	0.56 +/- 0.10
280	5	90	0.67 +/- 0.39
280	5	95	0.66 +/- 0.75
280	10	90	1.27 +/- 0.15
280	10	95	1.23 +/- 0.25

Table 2. Average etch rates for conditions in the 2^3 factorial design.

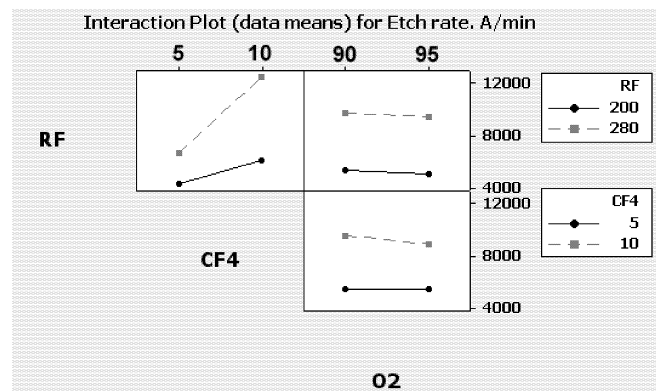

Fig 3. RF-CF_4 interaction and main effects plots for the 2^3 DOE (α = 0.05).

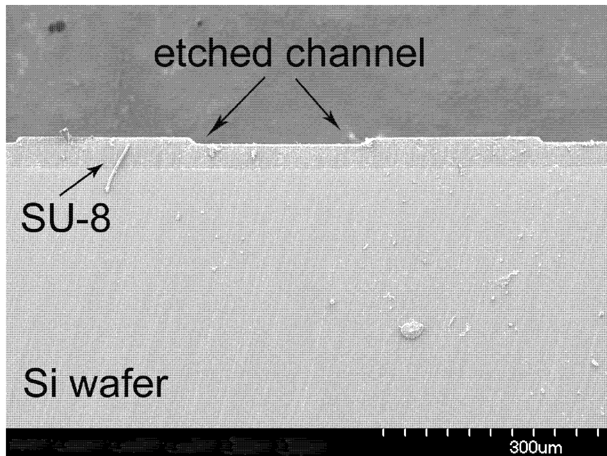

Fig 4. Scanning electron microscope image of cross section of etched channel in SU-8 2050 using a dry film mask (150X).

B. SU-8 surface conditions after RIE

The arithmetic average surface roughness for SU-8 doubled after RIE. The surface roughness of a virgin SU-8 surface was measured at 123nm, and an etched surface was measured at 213nm. A two-sample t-test gave a p-value = 0.108 at the 95% confidence level ($\alpha = 0.05$), and this result suggested that there was little statistical difference in the arithmetic average surface roughness of the virgin and etched SU-8 surfaces.

Water droplets on virgin SU-8 surfaces had a contact angle of ~80º, and etched surfaces had a contact angle of ~12º. During etching, F formed radical sites on the SU-8 polymer structure [11]. Water may have been attracted to these radical sites, thus making the etched SU-8 surface hydrophilic.

C. SU-8 wafer bonding

The UV exposure intensity, rather than bonding time, appeared to have a greater effect on SU-8 bond strength. The maximum strengths for a fully polymerized SU-8 surface bonded to a partailly polymerized blanket SU-8 2050 surface were ~1100psi for 50mJ/cm^2 exposure, ~1250psi for a 70mJ/cm^2 exposure, and ~280psi for a 100mJ/cm^2 exposure. It may be possible that remaining polymer solvent in the partially polymerized blanket SU-8 may have aided the bonding process, since no bonding occurred when attempting to bond two fully polymerized SU-8 surfaces. Final crosslinking of the blanket film may have occurred during the bonding process.

A channel ~25μm deep was etched into fully polymerized SU-8 using the RIE baseline process, and bonded for one hour to a partially polymerized SU-8 layer to create an internal channel (Figure 5). The sidewall angles for the RIE channels varied from 35º to 71º and had rounded corners at the bototm of the channels. These features may actually promote fluid flow since right angle corners at the bottom of a channel may create regions where fluid may remain stagnant. Channels in SU-8 were also created via photolithography and bonded to partially polymerized SU-8 (Figure 6).

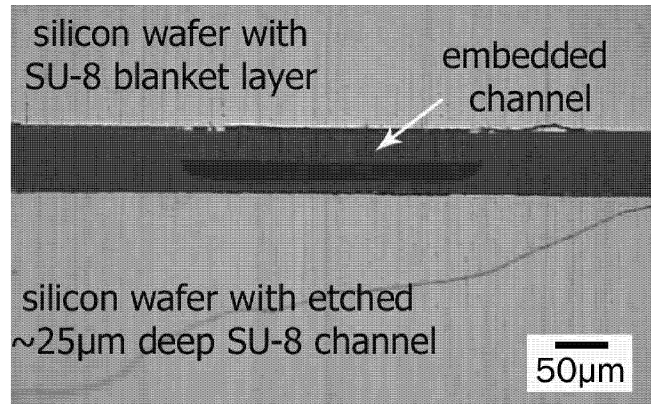

Fig 5. Optical image of cross section of RIE defined SU-8 channel and bonded to a blanket SU-8 film on a Si wafer (200X).

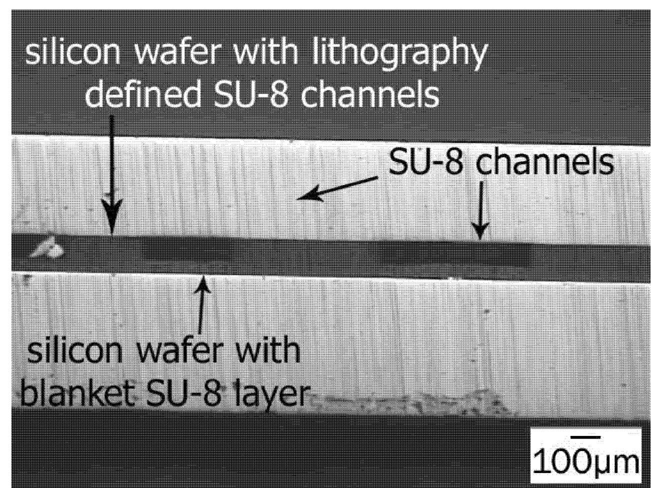

Fig. 6. Optical image of cross section of lithography defined channels and bonded to a blanket SU-8 film on a Si wafer (50X).

IV. CONCLUSION

Fully polymerized SU-8 was dry etched using RIE with etch rates of ~1.2μm/minute. Channels for fluid flow were formed in SU-8 using polyimide tape and dry film masks. Internal channels were created by bonding an RIE patterned SU-8 layer to a partially polymerized blanket SU-8 layer. Bond strengths as high as 1250psi were attained by bonding fully polymerized SU-8 to a partially polymerized SU-8 layer that was exposed to <100mJ/cm^2 UV light. The blanket film may have finished cross-linking during the bonding process.

The RIE process doubled the arithmetic surface roughness of the SU-8 whilst making the SU-8 surface hydrophilic and possibly enhancing fluid velocity in the SU-8 channels. The sidewall angles were >90º and had rounded corners, which may also enhance fluid flow or fluid velocity.

Future plans for this project include introducing a metal mask for the RIE process to etch channels of different widths or etch sophisticated patterns on SU-8. Different fluids such as water, serum or other biological fluids will be injected into the internal SU-8 channels formed by RIE and wafer bonding to measure fluid velocities and determine the effects of SU-8 surface roughness after RIE. Finally a prototype microfluidic device will be created that will incorporate all the process refinements in vertical electrodes formation, RIE, wafer bonding, and enzyme column immobilization.

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