

# Polymer (SU-8) Optofluidic Device with Embedded Hydrogel Oxygen Sensing Elements

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**Abstract** — The SU-8 photopolymer is an excellent alternative for traditional materials used in fluidic devices such as glass- or silicon-based inorganic materials. We describe a new chemically-anchored, lithographically-patterned hydrogel sensing element embedded in a completed SU-8 fluidic channel structure after the channel bonding process. Polyethylene glycol (PEG) hydrogel films incorporating oxygen-responsive ruthenium-complex fluorophores are grafted and patterned by photopolymerization of solution-phase precursors on the modified SU-8 channel surface. The optofluidic sensing platform with this embedded sensing elements exhibited excellent performance in measuring dissolved oxygen content. A unique advantage of this method is the selective growth of various functional films *in situ* by sequentially injecting different precursors. This fluidic sensing platform will be a viable component for both electrochemical and optical bioanalytical assays.

## I. INTRODUCTION

There is growing interest in developing fluidic devices for bioanalytical purposes [1]. Choosing the suitable materials for fluidic channels and matrix sensing elements within the fluidic channels becomes a main technical issue in this area. Due to the high flexibility and versatility in the process development of fluidic channel devices, the epoxy-based, high-aspect ratio photoresist SU-8 is attracting attention as the main structural material of fluidic devices [2]. For bioanalytical assays to be conducted in a seamless and integrated way, the fluidic channel wall (i.e. SU-8 surface) should be amenable for modification. Such modifications include the attachment of functional elements or chemical treatment to minimize possible biofouling issues. Forming functional elements (e.g. sensing elements) before completing the fluidic channel, however, is challenging since these elements (mostly biological organic substances) do not tolerate the high temperature during the wafer bonding process to complete the channel cavity structure.

Research in this area, as a result, is currently focused on adding functional elements after completion of the fluidic channel structure to avoid those harsh conditions during the wafer bonding process. For example, an integrated in-device electrochemical glucose sensor has been reported. Photodefinable poly(vinyl alcohol)-based membranes, which serve as the matrix for glucose oxidase (GOD), were patterned and polymerized within side pockets or around posts after the channel bonding process [3]. The effect of harsh conditions required was circumvented by this approach.

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Previously we described a surface modification method adapted from a photografted polymerization procedure utilizing a covalently linked initiator, for controlling the hydrophilicity of an SU-8 surface [4]. This was later tailored for immobilizing optical dye-containing films on planar SU-8 surfaces [5]. Here, we report our recent results on the fabrication and characterization of fluidic sensing platforms with embedded hydrogel sensing elements in a completed channel cavity structure.

## II. EXPERIMENTAL

### A. SU-8 device preparation

Glass microscope slides were cleaned by a standard method and dehydrated before coating with SU-8. A small amount of SU-8 2050 was spin-coated on the glass slide to produce an SU-8 layer of about 75  $\mu\text{m}$  thickness. This SU-8 layer was soft-baked, exposed to UV light, and then hard-baked. A second SU-8 layer was spin-coated at a thickness of 125  $\mu\text{m}$ . This second layer was used as the main material for the channel wall structure. It was first soft-baked to release any internal stress before being exposed to UV light in the presence of a photomask. After UV flood exposure, the material was post-baked. The channel structure was developed in propylene glycol methyl ether acetate (PGMEA)

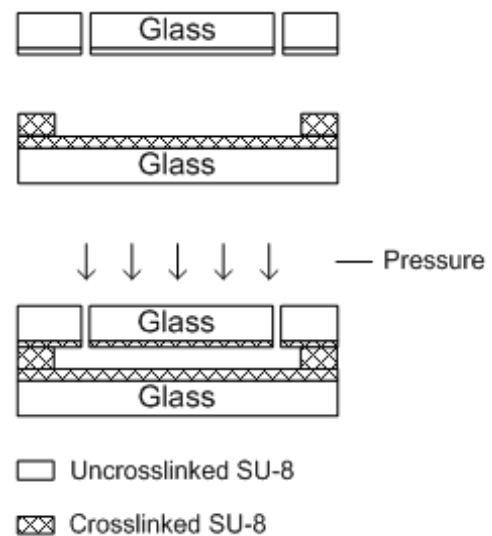
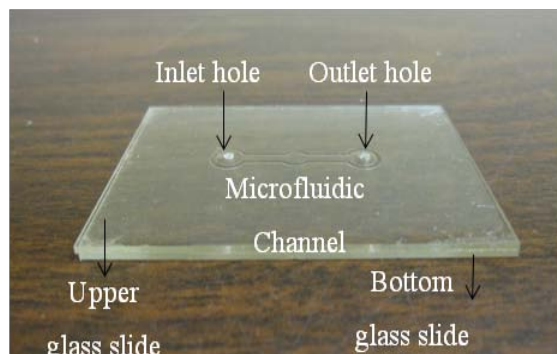
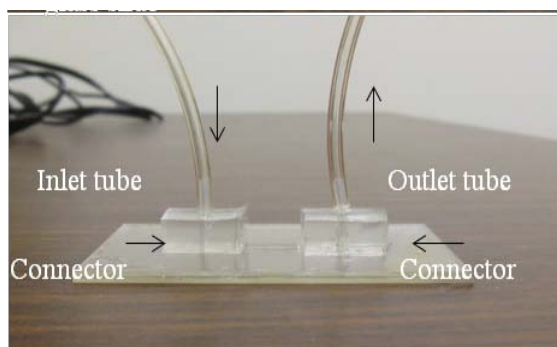


Figure 1. Bonding procedure of SU-8 fluidic channel structure.

and then rinsed with isopropanol. An identical glass slide as a cover substrate was cleaned and dehydrated with the same procedure. A small amount of SU-8 2050 diluted with SU-8 thinner was spin-coated and baked to produce a 20  $\mu\text{m}$  thick uncrosslinked SU-8 layer. Fluidic inlet and outlet ports were drilled on this cover substrate.



(a)



(b)

Figure 2. (a) A bonded SU-8 fluidic channel. (b) A completed SU-8 channel device with inlet and outlet tubes).

As shown in Figure 1, the glass substrate with the patterned, fully-cured SU-8 layer and the cover substrate with uncrosslinked SU-8 layer were placed on a hotplate. The glass transition temperature ( $T_g$ ) of uncrosslinked SU-8 is about 55°C [6]. If the bonding temperature is too low, it is hard for the SU-8 layers to bond with each other. On the other hand, if the bonding temperature is much higher than the glass transition temperature, the bonded SU-8 channel may be blocked if uncrosslinked SU-8 flows into the channel. We optimized the bonding temperature to be slightly higher than glass transition temperature of SU-8. It is considered that at that temperature, the SU-8 layer seems to be pliable to contact with each other and therefore most areas of the channel structure are bonded. External pressure was applied to help to make a tight contact. Finally, flood exposure through the transparent glass slide and a post-baking step were conducted to allow the uncrosslinked SU-8 layer to be polymerized. Figure 2 shows the SU-8 fluidic channel device.

### B. Hydrogel patterning

Photoinitiator solution saturated with  $\text{N}_2$  gas was injected into the completed channel and exposed to UV light as shown in Figure 3. The initiator was grafted on the channel wall surface to serve as the active site for anchoring polyethylene glycol (PEG) membrane. The details of the underlying chemical reaction principle were reported in our previous publication [4]. The modified channel was washed thoroughly with ethanol to remove any free initiators left in the channel. Later, an  $\text{N}_2$ -saturated hydrogel precursor solution (mixture of methacrylated PEG as a matrix material and ruthenium fluorescence complex as a detection agent) was injected into the SU-8 channel. A chemically-anchored, 1 mm diameter cylindrical structure of PEG-rich hydrogel was formed after the precursor solution within SU-8 channel was exposed to UV light with a mask, resulting in formation of a densely crosslinked, patterned hydrogel network. Any unpolymerized PEG macromonomer and untrapped ruthenium complex inside the channel were later washed with D.I. water.

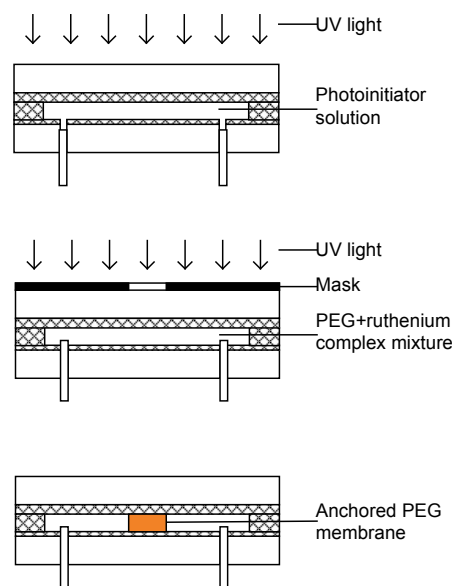


Figure 3. Fabrication sequence of chemically-anchored, lithographically-patterned hydrogel optical sensing element (polyethylene glycol + ruthenium fluorescence complex) within a photopolymer (SU-8) channel structure.

### C. Instrumentation

The setup for dissolved oxygen measurement is depicted in Figure 4. A blue LED (470 nm peak wavelength) was used as the excitation light source of ruthenium complex. The excitation light was delivered to the sensing element and the fluorescence emission was detected by illumination and reading fibers, respectively, included in a reflection probe. All fluorescence intensity measurements were conducted with a spectrofluorometer. A long wavelength pass filter (>540nm) was set to eliminate the strong blue excitation wavelength peak.

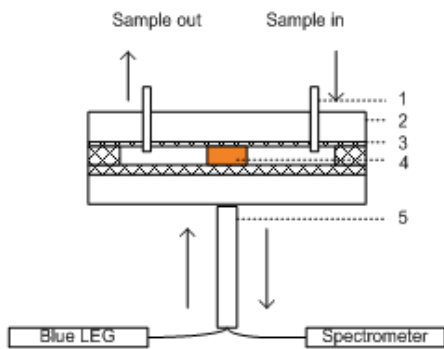


Figure 4. Measurement setup for SU-8 optofluidic channel sensor. (1) PFA tubing, (2) Glass slide, (3) Cross-linked SU-8, (4) PEG-based hydrogel, (5) Reflection probe.

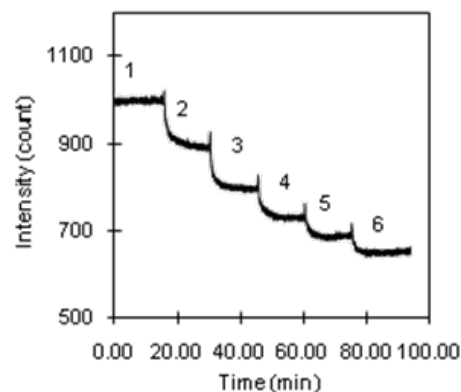
### III. RESULTS

The SU-8 channel was first filled with  $N_2$ -saturated deionized water before taking any oxygen measurements. Once the optical signal became stable, a series of deionized water samples with different dissolved oxygen concentrations were sequentially injected into the SU-8 fluidic channel with a syringe. Immediately after injecting each solution, the distal ends of plastic tubing at inlet and outlet ports were capped with fittings. Completely sealing the SU-8 fluidic channel minimized oxygen interference from the surrounding atmosphere since all parts and materials used to fabricate the SU-8 fluidic channel have low oxygen diffusion coefficients.

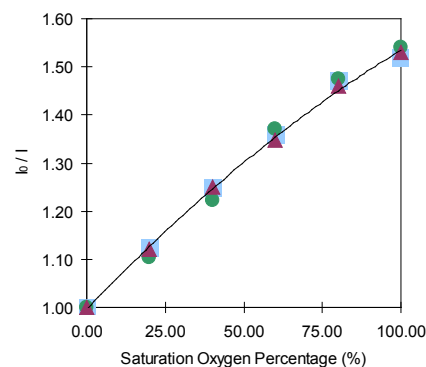
An obvious fluorescence emission difference (609 nm peak) was observed, as shown in Figure 5. The intensity signal decreased significantly when the sensing element was exposed to  $O_2$ -saturated water. This change was caused by the quenching behavior of ruthenium complex toward oxygen molecules, which demonstrates the optical sensitivity of the sensor toward the dissolved oxygen.

The peak fluorescent intensity (609 nm) decreased with the increase of the dissolved oxygen content steadily. As shown in Figure 6 (a), typical response time was about 5 minutes to reach the steady-state values. The cylindrical hydrogel structure remained in the middle of the SU-8 channel without physical shape change or migration, indicating the

successful anchoring of the functional element within the channel structure. Based on the fluorescent intensity data collected from three measurements, the Stern-Volmer relationship, which is a common method to evaluate the optical sensor based on emission quenching, is plotted in Figure 6 (b). It demonstrated a similar sensitivity as our preliminary data obtained with PEG films on plain SU-8 surfaces [5].



(a)



(b)

Figure 6. Double-chamber sensor characterization. (a) Images of the oxygen sensor during the two electrolytic bubble phases. (b) The results of (a) plotted on a Stern-Volmer relationship obtained with sample solutions with different oxygenation conditions.

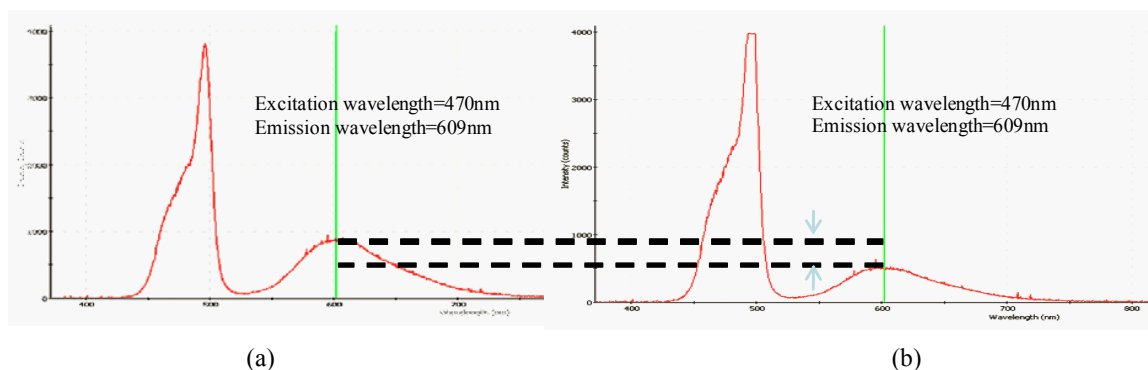


Figure 5. Oxygen quenching behavior of ruthenium complex entrapped in a PEG-rich hydrogel element patterned within a SU-8 channel. (a) 0%  $O_2$ -saturated deionized water. (b) 100%  $O_2$ -saturated deionized water.

#### IV. CONCLUSION

A fluidic, in-device optical oxygen sensor was successfully fabricated by integrating PEG-based sensing elements within completed SU-8 channels. The hydrogel precursor solution was lithographically patterned post-bonding and anchored inside SU-8 channel through photoinduced technique. Good performance of the sensing element was obtained. Therefore, the feasibility of forming viable sensing elements that are chemically anchored, lithographically patterned, and formed after SU-8 channel bonding process was confirmed. A further study is in progress to utilize this sensing element for immobilizing enzymes for bioanalytical applications such as glucose determination.

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