

# Inkjet printing of polymeric optical resonators for multi-gas sensing

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**Abstract**—Inkjet printing of polymer microdroplets combined with plasma etching is used to fabricate on-chip high-Q optical microresonators for gas sensing. Devices made of three different polymers (SU-8, polystyrene and polyhexamethylene biguanide) are characterized with carbon dioxide and using vapors of isopropyl alcohol and water.

**Index Terms**—Whispering gallery mode resonators, Optical resonators, Inkjet printing, Optical polymers, Gas sensor, Microfabrication

## I. INTRODUCTION

Portable gas sensors are needed for diverse applications including air quality monitoring, hazard detection in the mining and petroleum industries, and disease detection [1]. Among the different gas detection schemes, polymer matrices have shown promising performances in detecting different gases and volatile organic compounds (VOCs) as alterations in material properties occur upon gas absorption. While individual polymers lack selectivity to a specific gas, arrays of sensors comprising of different polymers are capable of analyte concentration retrieval with a mix of gases [2].

Optical gas sensors based on microresonators are useful in measuring these material property changes such as swelling and refractive index variations [3]. In contrast to electronic gas sensors, they do not necessitate heating and can be conducted over extensive lengths of optical fiber. For instance, concentrations within a mix of gases were recovered with an array of Fabry-Perot cavities filled with different polymers [4]. Greater sensitivities were achieved in detecting gases with whispering gallery mode resonators in the form of suspended polymer microdisks [5]. Inkjet printing of polymer materials was investigated to fabricate both suspended optical resonators [6] and electronic gas sensors [7]. This fabrication technique paves the way for a multi-polymer resonator array for gas sensing on a single optical chip.

Herein, we present the characterization of microdroplet resonators made out of three polymers as gas sensors reporting sensitivities and limits of detection.

## II. FABRICATION AND METHOD

The fabrication process of whispering gallery mode resonators made of a polymer using inkjet printing is illustrated in Fig. 1 a). The polymer inks are SU-8 in cyclopentanone

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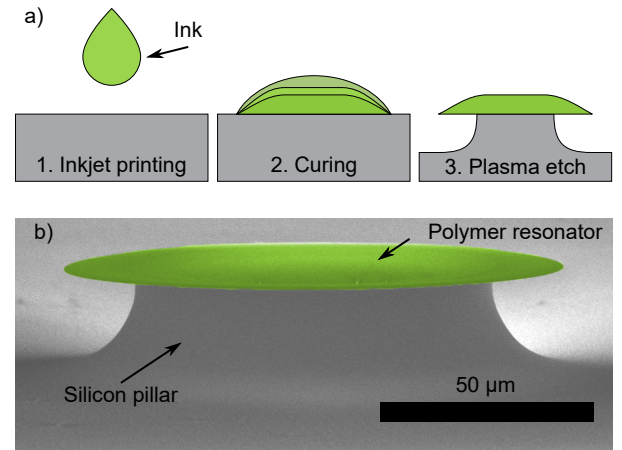


Fig. 1. a) The fabrication process initiates with inkjet printing of the polymer ink (step 1), followed by curing (step 2), and concluding with plasma etching of the silicon substrate (step 3). b) Scanning electron micrograph depicting a device where a polystyrene resonator in green false color resides on top of a silicon pillar.

(14.3 %w/w), polystyrene (PS) in propylene glycol methyl ether acetate (PGMEA) (9.9 %w/w) and polyhexamethylene biguanide (PHMB) in water (14.4 %w/w). The ink concentrations are chosen to be printable. Microdroplets are deposited on a cleaned silicon substrate using a Microdrop printer equipped with a piezoelectric head featuring a 100  $\mu\text{m}$ -diameter opening (step 1). Following the microdroplets deposition, SU-8 droplets undergo a baking and UV exposure process, while PS and PHMB droplets are left to dry at ambient temperature (step 2). Finally, the silicon substrate undergoes etching via an inductively coupled plasma of  $\text{SF}_6$  to release the edges of the microdroplets (step 3).

Gas sensitivity measurements are conducted following the established setup and methodology outlined in prior literature [4]. Isopropyl alcohol (IPA) and water ( $\text{H}_2\text{O}$ ) vapors are generated using a blubber, while carbon dioxide ( $\text{CO}_2$ ) is sourced from a gas cylinder. Both the gas and device temperatures are maintained at 30  $^\circ\text{C}$  [8]. Various concentrations of analytes are introduced onto the microdroplets, with concurrent recording of the spectral positions of the resonance peaks. The optical coupling is performed with a tapered optical fiber. Linear regression analysis of the peak displacements against concentration yields the sensitivity of

the specific polymer-analyte pair.

### III. RESULTS AND DISCUSSION

Figure 1 b) shows an example of a final device comprising a polystyrene microresonator on its silicon pillar. The printed microdroplets exhibit an approximate diameter of  $100\mu\text{m}$ , correlating with the size of the printer head opening, and a thickness around  $1\mu\text{m}$  at the edges. The different undercut are approximately  $15\mu\text{m}$  which is sufficient to limit optical mode leaking into the silicon pillar.

Optical characterization is conducted via transmission optical spectra analysis. Figure 2 a) depicts transmission spectra of the fabricated WGM resonators, revealing Q-factors of  $1.9 \times 10^5$ ,  $1.6 \times 10^6$  and  $8.4 \times 10^3$  and for SU-8, PS and PHMB respectively.

Sensitivities of the resonators to different analytes are reported in Fig. 2 b) and span two orders of magnitude with water exhibiting the highest sensitivity with  $0.25\text{ pm/ppmv}$ . As this technique is based on gas absorption, it is expected that the analyte with the highest saturation pressure, carbon dioxide, has the lowest sensitivity for all polymers.

The limit of detection (LOD) is defined as three times the noise over the sensitivity, with the noise derived from the peak displacement in steady state. From Fig. 2 c), we observe that the noise is lower for SU-8 and PHMB, thereby altering the performance order among the polymers. For the water-PHMB pair, the LOD is calculated to be  $14\text{ ppmv}$ .

Numerous factors influence the sensitivity and the LOD of this device. For the sensitivity, the degree of underetching dictates the extent of swelling [5]. Polymer conditioning such as UV and thermal curing alongside plasma treatment will also influence the performance [9]. Lastly, the ink formulation and dimension of the printer head opening affect the microdrop shape with parameters such as the edge contact angle and the resonator diameter. For the LOD, external factors influence the signal noise like temperature, pressure and coupling variations. The performance can be improved by designing the microresonator to become insensitive to temperature [8].

Given the distinct sensitivities observed for each polymer-analyte pair, it can become feasible to deduce the gas concentration of the three analytes based on the resonance peak shift of the polymer microresonators. Consequently, this platform offers the potential for multi-gas sensing. Further exploration of alternative polymers is required to attain greater differentials in sensitivities among the analytes, thereby enhancing performance.

### IV. CONCLUSION

The combination of inkjet printing of polymers with high Q-factor optical microresonators enables the creation of a multi-gas sensor housed on a single optical chip. Through the fabrication and characterization of devices comprising three distinct polymers, their respective gas sensing capabilities for three analytes were measured with limits of detection as low as  $14\text{ ppmv}$ . This platform demonstrates the feasibility of integrating multiple sensing materials onto a single chip.

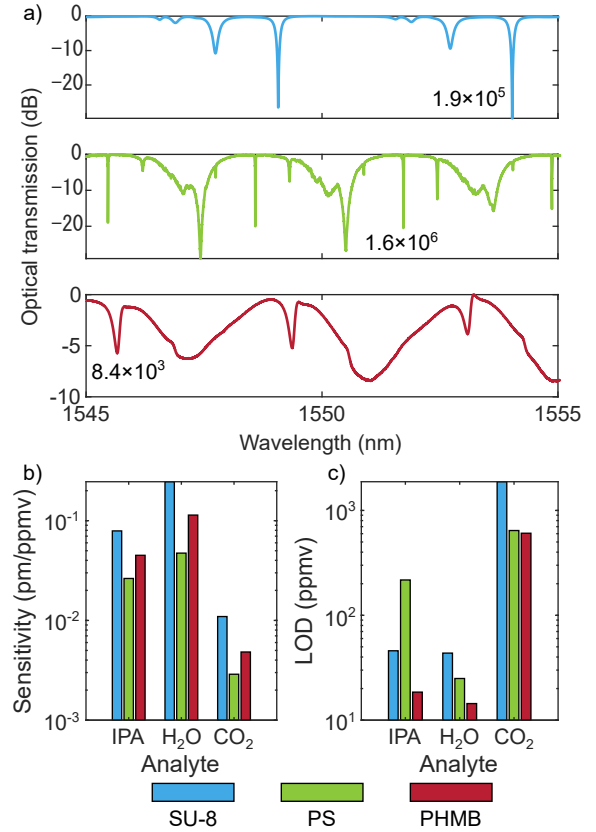


Fig. 2. Experimental results. a) Optical transmission spectra illustrating high-Q resonance peaks observed in SU-8, PS, and PHMB microresonators. b) Sensitivities and limits of detection (LODs) of the three devices with isopropyl alcohol (IPA), water, and carbon dioxide (CO<sub>2</sub>).

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